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"Optimization and Characterization of Ni-B Coating of Using RSM Method"

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Abstract: It is unique properties, the electroless method is used as an effective and important technique to create uniform coatings. Among the coatings that can be made by electroless process is Ni-B coating due to its excellent properties such as good corrosion resistance, high wear resistance, high hardness, favorable oxidation resistance. In this article, after introducing nickel-boron electroless coatings and examining the microstructure and wear rate, recent developments in strategies to improvement in corrosion resistance, tribological properties, and the use of nickel-boron electroless coatings as electrocatalysts. In this study operating parameters are optimized by using RSM method. operating parameters considered for the Reinforcement, temperature and frequency. This paper summarized, the parametric influence of Reinforcement, temperature and frequency on wear rate of Ni-B with ZrSi coating. Response surface methodology (RSM) is used for modelling and optimization. ANOVA has been carried out to identify importance of the operating parameters on the performance characteristics considered. Further the verification experiment has been carried out to confirm the performance of optimum parameters. The results from this study will be useful for selecting appropriate set of process parameters to Ni-B coating has been selected. The analysis of variance (ANOVA) has been used to determine effect of each parameter on wear rate so Finally, the confirmation test has been carried out to compare the predicted value of wear rate with the experimental value.
Keywords: Tribometer, RSM Method, Optimization, Wear rate

I. INTRODUCTION

In any engineering part, first the surfaces are subjected to the frictional, thermal, mechanical, chemical as well as electrochemical interaction which results in damage to the components in the industry. This damage cannot be recovered if the tribological and corrosion phenomenon is not controlled properly. By the application of appropriate surface technology the damage can be prevented or at least delayed. So, surface coating technology is most efficiently used to increase hardness, wear resistance, corrosion resistance and resistance at high temperature without altering the properties of substrate. Modern researchers have adopted a new form of coating which is called composite coating. Normally the composite material includes different types based on matrix materials such as metal matrix composites, ceramic matrix composites, polymer matrix composites and carbon matrix composites. Among them metal matrix composite coating is popularly used in the industry for its versatileness

Coatings produced by chemical reduction method are increasingly used in technical applications. A great advantage of the coatings obtained with this method is the possibility to deposit them on elements with complicated shapes and made from various materials. Among the coatings obtained with the chemical reduction methods, the most common are nickel coatings, owing to their advantageous properties, like high hardness and resistance to friction-induced wear, as well as a resistance to corrosion. The properties of nickel coatings produced by chemical reduction method can be modified by adding during the plating process various chemical compounds as reducing agents. The reducing agent's type determines the chemical composition of the coating. An electroless nickel plating process in a bath containing sodium hypophosphite as reducing agent will produce a Ni-P alloy coating. The use of boranes will produce Ni-B alloy coatings

II. EXPERIMENTATION

A. *Methodology of Experiment*

Some parameters like reinforcement, temperature and frequency etc. play an important role in minimum wear rate. So as to overcome the existing problems, few optimization techniques have to be incorporated. Based on the mentioned parameters the following study was conducted to achieve the objective. Once the root cause of the problems which impacted the wear rate was identified and objectives were set to overcome the problem. Based on the observation, RSM method was followed to design the experiment to study the major contributing factors. Minitab-19 software was used to optimize the DOE using RSM techniques

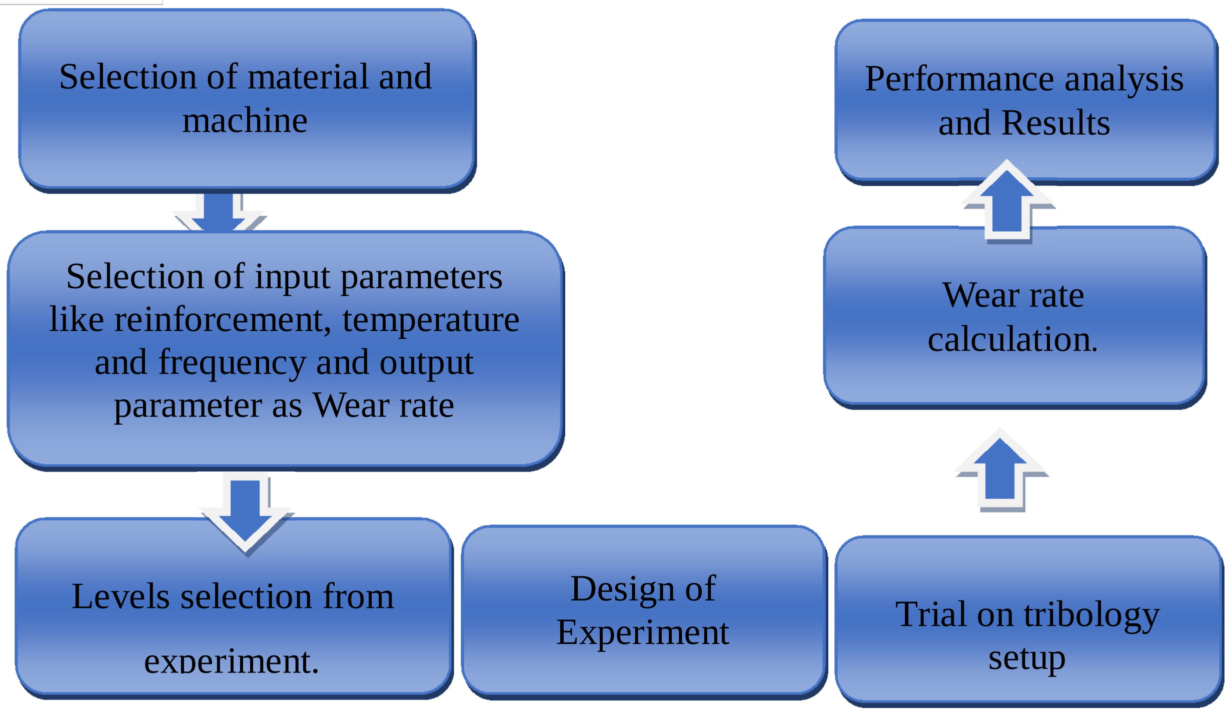

Fig.1 Flow Chart of Methodology.

Reinforcement, temperature and frequency were identified as the main factor affecting the coating process. Based on the observation, RSM method was followed to Design the experiment to study the major contributing factors. Minitab software was used to optimize the Design of Experiments using Taguchi techniques. Validation/Analysis of the contributing factors (process parameters) for laser cutting was done with the help of ANOVA tool (Analysis Of Variance).

To get the perfect result of the Ni-B coating process by using the Tribometer we need to find the correct parameter setting. Until now, so, it is important to find the best parameter setting before start the oating process in order to achieve the maximum result in its Wear rate. In this work, MS material is to be used as the work piece material.

B. Experimental Machine Selection

All the experiments were conducted at Govt. college of engineering, Aurangabad, M.S, India

Figure 2: Schematic Setup of LTR

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Table 1: Specifications of LTR Setup

Make Model	Ducom Ltd., Banglore, India
Upper Specification	Pin(dia. × 1)- Φ4×15mm, Φ6×15mm, Φ8×15mm, Φ10×15mm. Pin Rectangular (l×b×h)-4×6×15 Pin Square (l×b×h)- 4×4×15mm, 6×6×15mm, 8×8×15mm. Ball- Φ10mm
Lower Specification	Rectangular Block (l×b×h)- 40×40×5, 30×30×5, 20×20×5mm
Lower Specification	EN-31 Steel
Lower Specification Hardness	60 HRC
Stroke Length Range	10, 20 30 fixed.
Load Range	5 to 100 N (In step of 5N)
Temperature Range	Ambient 200 to 200^o C, Ambient 200 to 200^o C (For Both Lubrication). Least count..-0.21^{o}C, Sensor: PT-100
Frequency (Speed) Range1	1-20Hz(1200rpm) Least count: 1rpm, Sensor, Proximity Sensor
Frictional Force	0.1-100N Least Count: 0.1N, Sensor- Piezo Sensor
Wear Measurement Range	±2 mm, with least count 1 micron
Water Supply	2-5 lpm Provision inbuilt for internal connection. Connect the tap water from outside while conducting heating test.
Power	230 V× 1 Φ×50Hz,8A(For Tester)

C. Materials and Methods

In case of the present study, Nickel-Boron (Ni-B) and ternary Nickel-Boron-Alumina (Ni-B-ZrSi). Mild steel substrates of dimension 20 mm x 20 mm x 5 mm were deposited upon. The primary bath composition used for depositing both the coatings is given in below Table. The Ni-B coating was reduced by sodium borohydride solution added just before the introduction of the substrate in the bath. Additionally to deposit the ternary Nickel-Boron-ZrSi composite coatings,. The Ni-B coating was reduced by sodium borohydride solution added just before the introduction of the substrate in the bath. Before adding the ZrSi to the coating bath these nano powders. These polished specimens underwent all the necessary cleaning process such as ultrasonic cleaning in deionised water, acid pickling as well as cleaning with ethanol to ensure a completely dry and contaminant free surface. To ensure that the deposition was carried out in a contaminant-free environment the glassware used were also properly cleaned and finally rinsed using deionised water before the commencement of the coating.

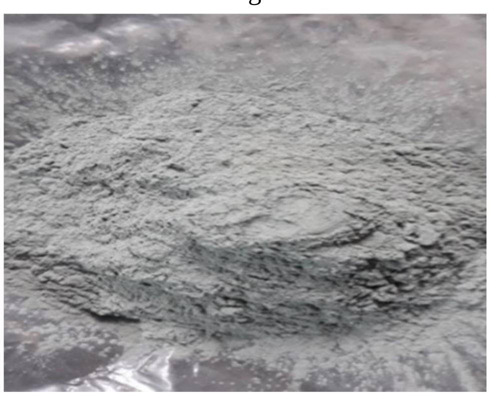

Fig 3. ZrSi Powder

III. RESULTS AND DISCUSSION

To get complete understanding of effects of input parameters reinforcement, temperature and frequency on output Wear rate, you usually assess signal to noise ratio or main effects plot for means. For this purpose, Minitab 19 statistical software has been used. It has been done. ANOVA has been conducted to find out effect of each parameter on the wear rate and linear regression model has been established to predict the values wear rate.

A. Experimentation

RSM is used to develop second-order regression equations relating response characteristics and process variables. A face centered CCD has three groups of design points :(a) two level factorial or fractional factorial design points (b) axial points and (c) center points. Center points are usually repeated 6 times to get a good estimate of an experimental error (pure error).

Table 2: Experimental matrix and Output response table

Run	Factor			Response
	Reinforcement (%)	Temperature (^oC)	Frequency (Hz)	Wear rate (mm^3/min)
1	2.0	45.00	4	0.0235
2	0.00	65.00	6	0.0255
3	0.00	65.00	2	0.0238
4	4.00	65.00	6	0.0229
5	2.00	65.00	4	0.0241
6	4.00	45.00	6	0.0225
7	2.00	55.00	4	0.0311
8	0.00	45.00	2	0.0302
9	4.00	55.00	4	0.0301
10	2.00	55.00	4	0.0308
11	4.00	65.00	2	0.0236
12	2.00	55.00	7	0.0247
13	2.00	55.00	2	0.0243
14	2.00	55.00	4	0.0312
15	4.00	45.00	2	0.0259
16	5.36	55.00	4	0.0276
17	2.00	45.00	6	0.0283

B. Analysis of Variance (ANOVA)

ANOVA is carried out to statistically analyze the results. ANOVA checks the values of R2 as it explains the ratio of the variability explained by the model to the total variability inherent inthe observation data of experiments. The F-value in the analysis compares model/factor variance with the residual variance (sum of square ratio). If the variance values are close to each other, then F-value is close to unity and it is less likely that the model/factor to have significant effect on the output response. Process variables having p-value < 0.05 are considered significant terms for the given response parameters.

Table 3: ANOVA table for Wear rate

Source	Sum of Squares	df	Mean Square	F-value	p-value	% Contribution
Model	1.790	9	1.989	3.05	0.0486	Significant
A- Reinforcement (%)	1.650	1	1.650	2.53	0.0142	18.56
B- Temperature (^oC)	2.923	1	2.923	4.482	0.0479	32.89
C- Frequency (Hz)	4.184	1	4.184	6.41	0.0297	47.08
AB	4.232	1	4.232	6.49	0.0290	
AC	4.805	1	4.805	0.74	0.4108	
BC	1.800	1	1.800	0.028	0.8714	
A^2	2.698	1	2.698	4.14	0.0693	
B^2	2.373	1	2.373	3.64	0.0856	
C^2	1.698	1	1.698	2.60	0.1377	
Residual	6.522	10	6.522			
Lack of Fit	4.444	5	8.887	2.14	0.2121	Not Significant
Pure Error	2.097	5	4.158			
Core Total	2.443	19				

Determination of the process parameters affecting the wear rate are done by carrying out ANOVA analysis on the response surface model. Based on the p-value of less than 0.05, Frequency is identified as the major influencing parameter contributing 47.08 % to performance measure, followed by

Reinforcement and Temperature that contributing 18.56 % and 32.89 % respectively. Graph 1 shows the 3D surface for wear rate variations in response to changes in process parameters. This indicates that the wear rate increases with increase in frequency and temperature.

C. Selection of Adequate Model

To evaluate for sufficiency of the model, lack of fit test has been performed for Wear rate. For the model to be fit, this test should indicate an insignificant lack of fit. Lack of fit test measures how well each of the polynomial models fit the data, by comparing the residual error to the pure error from replicated design points. Significantly larger residual error than pure error indicates that something remains in the residuals that can be removed by more appropriatemodelling. Lack of fit test for wear rate is shown in table 4 respectively. A p-value,greater than 0.05 nullifies the lack of fit of the model to the response data, and the model can be utilized for prediction of response parameter for 95% of a confidence interval. Model summary statistics give information about standard deviation, R^2, adjusted R^2. Generally, a model with a smaller standard deviation, R^2 closer to 1 is selected.

Table 4 Lack of fit test for Wear Rate

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Linear	1.165	11	1.059	4.37	0.2010	
2FI	8.623	8	1.078	4.44	0.1966	
Quadratic	2.875	5	5.751	2.37	0.3228	Suggested
Cubic	6.605	1	6.605	2.72	0.2407	Aliased
Pure Error	4.851	5	2.425			

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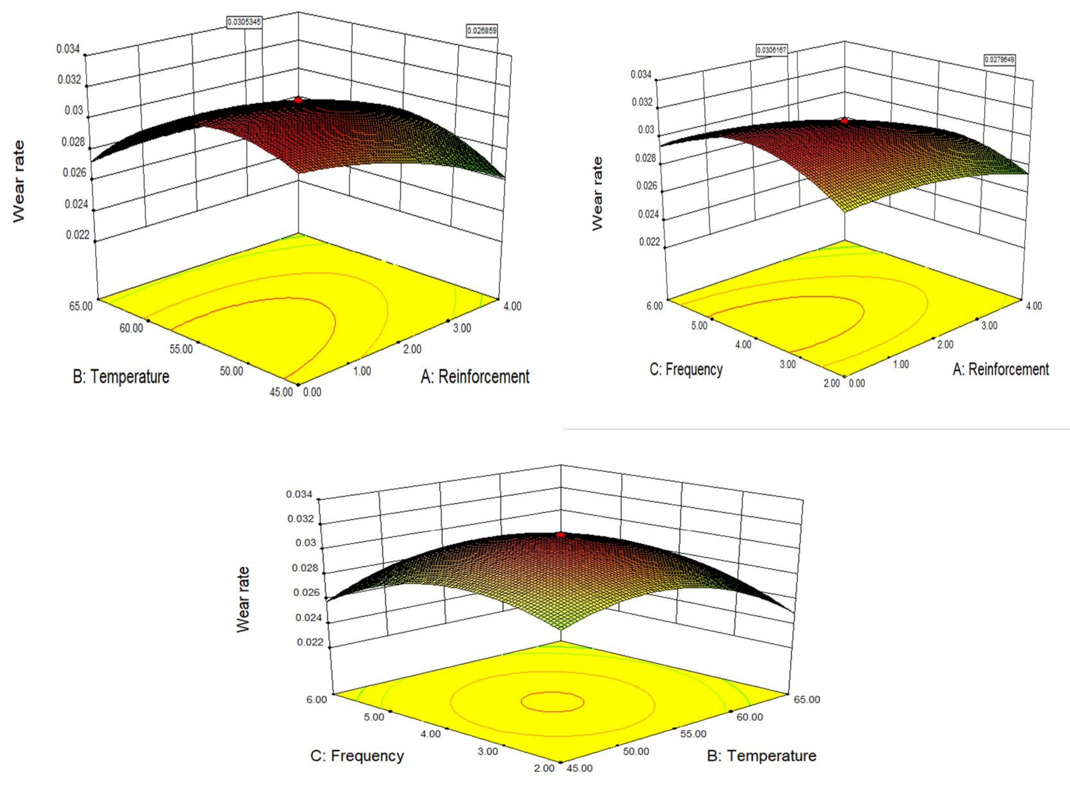
1) *Model Summary:* R^2 explains the degree to which input parameter explain the variation of output/predicted response. So, higher the R^2, more variation is explained by input parameters and hence better is the model. However, the problem with the R^2 is that it will either stay the same or increase with the addition of more parameters, even if they do not have any relationship with the outputresponse. To address this problem adjusted R^2 is used. Adjusted R^2 penalizes for adding parameters that do not improve existing model. The values of R^2 are always greater than or equal to adjusted R^2 and is shown in table 5. R^2 value equal to 00.9511 is in good agreementwith the adjusted R^2.

Table 5: Regression model summary for wear rate

Std. Dev.	2.554	R^2	0.9511
Mean	0.029	Adjusted R^2	0.9077
C.V. %	8.76	Predicted R^2	0.8632
		deq Precision	5.905

D. *Development of Regression Model for Wear Rate*

Regression model is developed using Design Expert v13 software. Substituting the experimental values of the parameters in regression equation, values for wear rate are predicted forall levels of study parameters. Graphical representation also shows that a predicted and experimental values of wear rate correlate with each other. Mathematical model for reinforcement, temperature and frequency is calculated using design expert v13 software and analysis of regression is carried out to obtain the predicted value of wear rate.



Graph1 3D plots for the Wear Rate

Difference between wear rate values calculated using regression equation and experimental values for each experience found less than 10%. Hence, we can say that the regression equation developed is valid. Graph 1 show the graphical representation of experimental and predicted values calculated using regression equation. CCD design and ANOVA analysis give
Mathematical model that predict the result nearly accurate.

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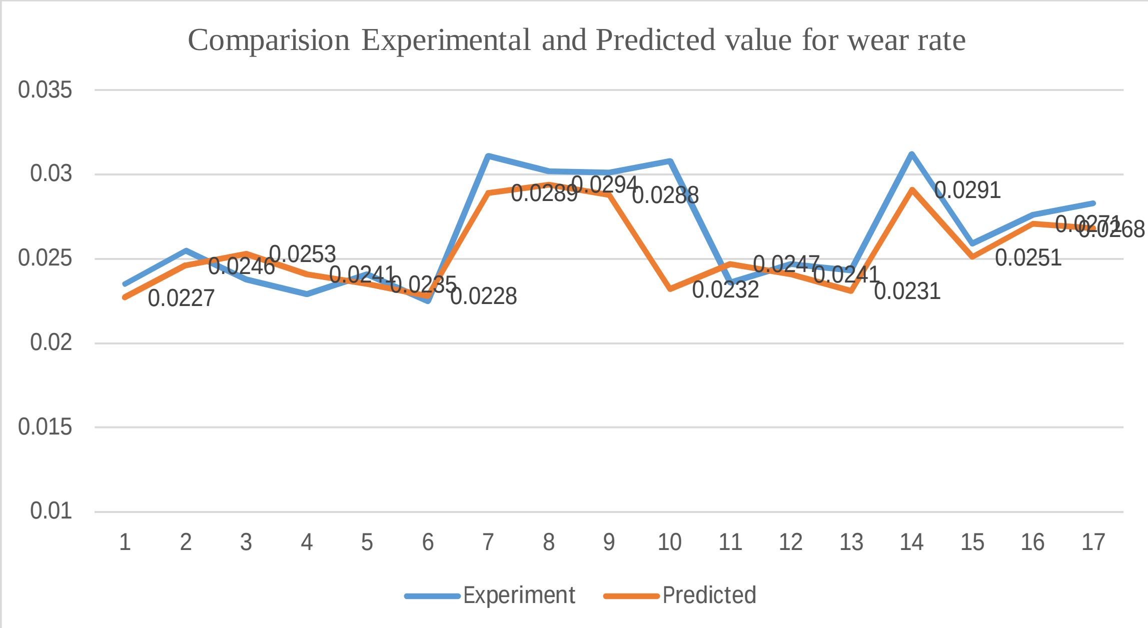

Graph 2 The comparison between experimental and predicted value of wear rate

E. Confirmation Experiment

To confirm the optimal result, confirmation test is conducted using the same experimental set up for reinforcement at 4,% temperature at 65°C and frequency at 2Hz. Results of the Confirmation experiment for wear rate is shown in table 6.

Table 6. Confirmation experiment result

Parameter	Predicted value	Experimental value	Error %
Wear rate (mm3/min)	0.0219	0.0222	1.36

Confirmation experiment is conducted by keeping parameters at optimum levels suggested by design expert v13 software and obtained wear rate value are compared with the predicted value given by the regression model. It can be seen that the difference between experimental result and the predicted result for wear rate is 1.36% respectively. This indicatethat the experimental value correlates to the estimated value.

IV. CONCLUSIONS

In this study the influence of operating parameters such as Reinforcement, Temperature, Frequency and their optimization for Ni-B with ZrSi has been studied by using RSM Method. Following conclusions are drawn.

1) The optimal solution obtained for Wear Rate based on the combination of wear parameters parameters and their levels is Reinforcement 4% (level 3), Temperature 65°C(level 3) and Frequency 2 Hz (level 1)
2) ANOVA results indicate that frequency plays prominent role in determining the Wear Rate. The contribution of Reinforcement, Temp and Frequency to the quality characteristics Wear Rate is 18.56%, 32.89% and 47.08% respectively.
3) ANOVA results indicate that contribution of reinforcement on Wear Rate is lower followed by Reinforcement and Load. Reinforcement is most dominant factor.
4) Value of Wear Rate is lower obtained in confirmation experiment. Hence, good quality of Ni-B with ZrSi can be achieved using suggested level of parameters by RSM method.
5) Values of Wear Rate calculated using regression model correlates with experimental values with error less than 10%. Hence the model developed is valid and experimental results of Wear Rate with any combination of operating parameters can be estimated within selected levels.
6) Regression equation has been successfully used to develop mathematical model for wear rate

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