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RESEARCH

Wear Performance Optimization of Electroless Ni-B Coating Using Taguchi Design of Experiments

The present study outlines the use of Taguchi parameter design to minimize the wear performance of electroless Ni-B coating by optimizing the tribological testing parameters. The tests are carried out in a multi- tribotester and the three parameters viz. load (L), speed (S) and time (T) are considered with three levels each. An L27 array is used to accommodate the three factors as well as their interaction effects. The Taguchi experiments gave the optimal combination of parameters L1S2T1 (50 N for load, 60 rpm for speed and 5 minute for time). Furthermore, a statistical analysis of variance reveals that both load and time have significant influence over the wear behavior of electroless coating. Also the interaction between load and speed and that between load and time influence wear quite significantly. The coating is characterized using scanning electron microscopy, energy dispersive X-ray analysis and X-ray diffraction analysis. The wear mechanism is also studied and found to be abrasive in nature.

Keywords: *sliding wear, Ni-B, electroless coating, Taguchi method, optimization.*

1. INTRODUCTION

The discovery of electroless coatings is credited to Brenner and Riddell [1] in the 1940s. Today electroless nickel has grown into a very substantial segment of the metal products finishing industry. Engineering applications for electroless nickel can be found in virtually every industry. Various physical characteristics of electroless nickel coatings, such as hardness, wear resistance, coating uniformity and corrosion resistance, as well as the ability to plate non-conductive surfaces make this a coating of choice for many engineering applications [2]. Hypophosphite reduced Ni-P coating [3-6] has already been widely accepted and the quest for achieving a superior hard and wear resistant surface has brought Ni-B coatings at the focus of research [7-19]. Electroless Ni-B plating is widely used in aerospace and automotive industries particularly due to their high hardness and hence splendid wear resistance [2]. Ni-B coatings are found to be harder than Ni-P coatings in as deposited phase [7]. With heat treatment, the hardness of Ni-B coating is found to increase even more [7-10].

The increase of hardness of Ni-B coating with heat treatment is generally attributed to the modification of deposit structure allowing the precipitation of Ni-B phases according to the Ni-B phase diagram [8].As harder materials generally encounter lesser wear, heat treated Ni-B coatings are found to be more wear resistant than the as deposited ones [7,11,12]. Moreover, Ni-B coating possesses a columnar structure, which is useful in retaining lubricants under conditions of adhesive wear [13]. Krishnaveni et al [11] have found that specific wear rate and friction coefficient of electroless Ni-B coating increases with increase in applied load in pin-on-disc arrangement. Scratch test by Delaunois and Lienard [8] point towards the fact, that heat treatment could also increase the adhesion between the Ni-B deposit and the substrate. Search of improved tribological properties has led to the formation of duplex coatings of Ni-P and Ni-B [7] and three component coatings of Ni-B-P [20, 21]. When tribological characteristics are of primary concern the design engineer must find innovative methods for integrating a base material having certain bulk properties with a properly functioning surface and electroless Ni-B coating has proved to be a good tribological material in that respect.

Tribology is vital to modern machinery involving sliding and rolling surfaces. Wear, an important aspect of tribology is often the limiting mechanism of device service life. Hence, except in some limited cases, wear is an unwanted phenomenon

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and needs to be minimized if not completely eliminated. The present study deals with the application of Taguchi method to determine the suitable tribological testing parameters in order to obtain minimized wear performance of electroless Ni–B coatings. Ni–B coatings are applied on mild steel (AISI 1040) specimens and then annealed at 350°C for 1 hour. The wear behaviour of the coated specimens is then evaluated by a multi-tribotester. Three testing parameters viz. load, speed and time are varied according to the Taguchi orthogonal design and the optimal combination of testing parameters is obtained based on minimization of wear. A confirmation test is performed to verify the optimal test parameters as predicted by Taguchi method. An analysis of variance is also carried out to observe the significance of factors and their interactions on the wear performance. The characterization of the coating is done with the help of scanning electron microscopy, energy dispersive X-ray analysis and X-ray diffraction analyser.

2. DESIGN OF EXPERIMENTS USING TAGUCHI METHOD

Taguchi techniques were developed by G. Taguchi [22-24]; these techniques have been utilized widely in engineering analysis to optimize the performance characteristics with the combination of design parameters. Taguchi technique is also a powerful tool for the design of high quality systems. To achieve quality improvement, Taguchi pioneered the use of robust parameter design. Robust parameter design is an engineering method for product and process design that focuses on minimizing variation and/or sensitivity to noise.

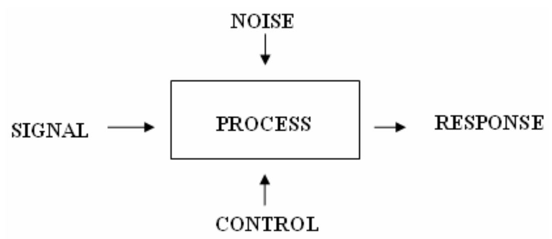


Figure 1. General model of a process/system

In general, a system or process (as seen in Fig. 1) can be visualized as a combination of machines, methods, people and other resources that transforms some input (substrate materials, chemicals, machines, equipments, energy, manpower, etc.) into an output (deposited mass, compositions, structures, properties) that has one or more observable responses. There are some process variables called noise factors, which are actually disturbances that cause the system response to shift from the specification. These factors are likely beyond the designer's control, such as manufacturing tolerances, environmental conditions, errors in measuring instrument, human errors, etc. Control factors (load, speed, time, temperature, lubrication, etc.) can be controlled by the designer to compensate for noise factors that could significantly influence the system away from desired performance.

Design of Experiments (DOE) is based on the objective of desensitizing a product's performance characteristic(s) to variation in critical product and process design parameters. Through DOE, a series of tests are performed where preplanned changes are made to the controllable variables so that the reason for changes in the response can be observed and identified. When DOE is performed using Taguchi method, the latter generally relates the variability in the responses of a particular trial condition with the effect of the uncontrollable variables or noise. Taguchi method obtains the optimal condition by observing the reduction in variation of the results within a particular trial condition. Hence, to take into account the variation of results, Taguchi method uses the S/N ratio to identify the quality characteristics applied for engineering design problems. In the S/N ratio, signal is interpreted as mean while noise is interpreted as standard deviation. Thus, Taguchi method tries to reach optimality by maximizing the S/N ratio so that the effect of noise is minimized. The S/N ratio characteristics can again be divided into three categories based on objective of the experiment viz.: lower-the-better (LB), higher-the better (HB) and nominal-the best (NB). For the present case of minimization of wear, LB characteristic needs to be used. Moreover, Taguchi method employs a special design of orthogonal arrays (OA) to learn the whole parameters space with only a small number of experiments. Based on OA, the number of experiments which may increase the time and cost can be reduced. In the present study, an L27 OA is used to study the effect of tribo-testing parameters as well as their interactions on the wear performance of electroless Ni-B coatings. Furthermore, to know which of the testing parameters have a significant influence over the wear performance of Ni-B coating; analysis of variance (ANOVA) [25] is also performed. Finally, to verification and validation of the optimal condition obtained through OA design is carried out through a confirmation test and the improvement in the wear performance characteristics at the optimal condition is compared to the initial condition.

3. EXPERIMENTAL DETAILS

3.1 Coating development

Steel (AISI 1040) blocks of size 20 mm × 20 mm × 8 mm is used as the substrate for the deposition of electroless Ni-B coating. The dimension chosen for the substrates is in accordance with the counterpart in the multi-tribotester where the coated samples are to be fitted for the wear tests. The substrates are meticulously prepared so that after coating, the samples could be fitted precisely with its counterpart. After preparation, the substrates are cleaned with soap and water to clean off any foreign particles. A cleaning with acetone is then employed to clean any remaining organic products. The electroless bath is prepared by mixing the chemicals given in Table 1 with distilled water and in appropriate sequence. The chemicals are weighed on an electronic balance of resolution 10-5 g so that no compromise is made on the accuracy of the bath composition. Before coating, the substrates are activated by dipping in a palladium chloride solution kept at 55ºC. The activation is necessary to kick start the deposition process. After activation, the samples are immersed into the electroless bath maintained at 85ºC. The pH of the solution is maintained around12.5 by continuous monitoring with a digital pH meter. The deposition is carried out for about 2 hours and the thickness of the deposit is found to be around 25 micron as evident from the optical micrograph (Fig. 2) of the coating cross section. As heat treatment is found to have a positive influence on the hardness and wear resistance of electroless coatings, the coated samples are annealed in a box furnace (for 1 h) separately at a temperature of 350°C.

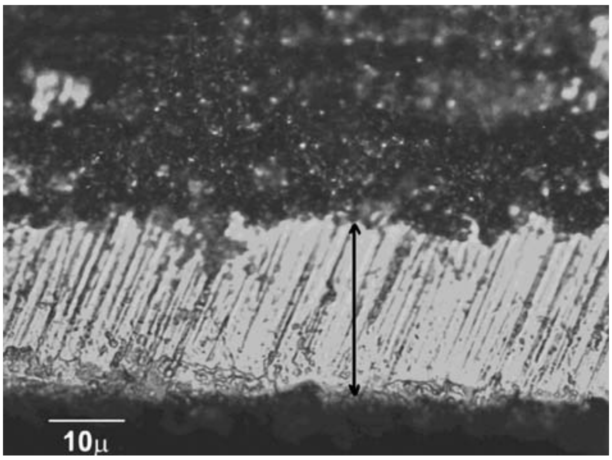


Figure 2. Optical micrograph of Ni-B coating cross-section

There have been several propositions regarding the reaction mechanism of electroless Ni-B coatings but the experimentally validated scheme [19] for the reaction mechanism of nickel boron plating consists of mainly three steps:

Reduction of nickel:

$$BH_4^- + 4H_2O \rightarrow B(OH)_4^- + 4H + 4H^+ + 4e^-$$

$$2Ni^{+2} + 4e^- \rightarrow 2Ni^0$$

$$BH_4^- + 2Ni^{+2} + 4H_2O \rightarrow 2Ni^0 + B(OH)_4^- + 2H_2 + 4H^+$$

Reduction of boron:

$$BH_4^- + H^+ \rightarrow BH_3 + H_2 \rightarrow B + \frac{5}{2}H_2$$

Hydrolysis of borohydride:

$$BH_4^- + 4H_2O \rightarrow B(OH)_4^- + 4H + 4H^+ + 4e^- \rightarrow B(OH)_4^- + 4H_2$$

It can be noted that wear characteristics of Ni-B coatings may be dependent on the surface roughness of the coating, which may again be dependent on the surface roughness of the substrate. However in the present case, the substrate roughness is assumed to be almost constant and hence not taken into account. Again achieving same roughness in all the substrates is very difficult, especially as each one is machined separately. Thus to solve this problem, a large number of substrates are prepared and their roughness (centre line average, R_a) are evaluated. The samples which showed small variation in roughness are used for the deposition of electroless Ni-B coatings. A surface profilometer, Talysurf (Taylor Hobson, Surtronic 3+) is used to measure the roughness values of the substrates prior to the coating. The R_a values of the substrates are found to lie in the range 0.2-0.3μm.

Table 1. Ni-B bath composition

Parameters	Values
Nickel chloride	20 g/l
Sodium borohydride	0.6 g/l
Ethylenediamine	59 g/l
Lead nitrate	0.0145 g/l
Sodium hydroxide	40 g/l

3.2 Choice of test parameters

Design parameters are the controllable factors that are suitably varied in order to obtain the desired performance. There are a large number of factors that can affect the tribological performance of electroless coatings. A review of the current literature revealed that the testing parameters viz. load (L), speed (S) and time (T) are easier to control and are also popular among researchers to control the tribological performance of electroless coating [5]. Hence these three factors are used as the main design parameters along with their interactions in the present study. The design factors along with their levels are shown in Table 2. The levels are chosen keeping in mind the low thickness of the coating otherwise wear may occur through the coating [5, 6].

Table 2. Design factors and their levels

Design factors	Unit	Levels		
		1	2	3
Load (L)	N	50	75[a]	100
Speed (S)	RPM	50	60[a]	70
Time (T)	min	5	10[a]	15

a : initial parameters

3.3 Response variable

Response variables are the requirements placed on the system's output. In the present study, minimization of the wear performance characteristics of electroless Ni-B coatings is the main objective. Hence, the response variable used to accomplish this study is the depth of wear in microns. The tribological testing parameters are optimized with the objective of minimizing the wear depth of electroless Ni-B coatings

3.4 Design of experiments

Design of experiments (DOE) is a technique to obtain the maximum amount of conclusive information from the minimum amount of work, time, energy, money, or other limited resource. The information generally comprises the relationship between product and process parameters and the desired performance characteristic. By learning and applying this technique, it is possible to significantly reduce the time required for experimental investigations. Orthogonal arrays (OA) are used to aid in the design of an experiment. The OA will specify the test cases to conduct the experiment. OAs also allow one to compute the main and interaction effects via a minimum number of experimental trials. Several standard OAs have been tabulated by Taguchi. The choice of a suitable OA design is critical for the success of an experiment and depends on the total degrees of freedom (dof) required to study the main and interaction effects, the goal of the experiment, resources and budget available and time constraints. Degree of freedom (dof) refers to the number of fair and independent comparisons that can be made from a set of observations. In the context of DOE, the number of degrees of freedom of a particular parameter is one less than the number of levels associated with the parameter. In the present case since each of the main factors is associated with three levels, the dof of each of the factors is two. Again the number of dofs associated with an interaction is the product of the number of dof associated with each main effect involved in the interaction. In the present case each interaction is associated with four dofs (2×2). Therefore the total dof for a three level design with three main parameter and three interactions is equal to eighteen (3×2 + 3×4). It is important to notice that the number of experimental trials in the OA must be greater than the total dof required for studying the effects. Hence, L_{27} OA, requiring twenty seven experimental runs is suitably chosen for the present case. The assignment of the factors and interactions to the columns of the array is done on the basis of the Triangular Table for 3-level OA [24] as suggested by Taguchi. The L_{27} OA together with the column assignments is shown in Table 3. The values in each cell of the main parameter columns (L, S, and T) in the array indicate their levels (1, 2 and 3). Again in case of interactions, two columns are assigned to a single interaction and the two cell values in a particular row indicate the levels of each of the factors involved in the interaction. The unassigned columns in the OA are kept for the errors terms.

3.5 Wear tests

A multi-tribotester apparatus (TR-25, DUCOM) is used for the wear test of electroless Ni-B coatings. The tests are carried out using a block on roller geometry. The values of the parameters are selected keeping in mind the low thickness of the coating which is found to be about 25-30μm (Fig. 1). The wear test is performed dry and at an ambient temperature of about 28°C with relative humidity of about 85%. A schematic diagram of the test rig is shown in Fig. 3. The Ni-B coated specimens are held stationary with the help of an attachment and made to slide against a rotating steel roller coated with titanium nitride of hardness 85 HRc. Wear is measured in terms of displacement (in microns) with the help of linear voltage resistance transducer. Wear displacement measured is actually the sum of the wear at specimen surface and that at the counter face surface. But as the hardness of the coatings is found to be around 70-72 HRc which is quite lower than the counter face material (85 HRc), the counter face material encounters negligible wear compared to the coated specimen.

Table 3. L_{27} Orthogonal Array with main parameters and interactions

	Column numbers												
Trial No.	1 (L)	2 (S)	3 (L×S)	4 (L×S)	5 (T)	6 (L×T)	7 (L×T)	8 (S×T)	9 -	10 -	11 (S×T)	12 -	13 -
1	1	1	1	1	1	1	1	1	1	1	1	1	1
2	1	1	1	1	2	2	2	2	2	2	2	2	2
3	1	1	1	1	3	3	3	3	3	3	3	3	3
4	1	2	2	2	1	1	1	2	2	2	3	3	3
5	1	2	2	2	2	2	2	3	3	3	1	1	1
6	1	2	2	2	3	3	3	1	1	1	2	2	2
7	1	3	3	3	1	1	1	3	3	3	2	2	2
8	1	3	3	3	2	2	2	1	1	1	3	3	3
9	1	3	3	3	3	3	3	2	2	2	1	1	1
10	2	1	2	3	1	2	3	1	2	3	1	2	3
11	2	1	2	3	2	3	1	2	3	1	2	3	1
12	2	1	2	3	3	1	2	3	1	2	3	1	2
13	2	2	3	1	1	2	3	2	3	1	3	1	2
14	2	2	3	1	2	3	1	3	1	2	1	2	3
15	2	2	3	1	3	1	2	1	2	3	2	3	1
16	2	3	1	2	1	2	3	3	1	2	2	3	1
17	2	3	1	2	2	3	1	1	2	3	3	1	2
18	2	3	1	2	3	1	2	2	3	1	1	2	3
19	3	1	3	2	1	3	2	1	3	2	1	3	2
20	3	1	3	2	2	1	3	2	1	3	2	1	3
21	3	1	3	2	3	2	1	3	2	1	3	2	1
22	3	2	1	3	1	3	2	2	1	3	3	2	1
23	3	2	1	3	2	1	3	3	2	1	1	3	2
24	3	2	1	3	3	2	1	1	3	2	2	1	3
25	3	3	2	1	1	3	2	3	2	1	2	1	3
26	3	3	2	1	2	1	3	1	3	2	3	2	1
27	3	3	2	1	3	2	1	2	1	3	1	3	2

Hence, the measured displacement can be taken as a representative of the actual wear depth encountered by the coating surface. It is worth noting that, in general wear is measured in terms of wear volume or mass loss. But in the present case, wear is expressed in terms of displacement or wear depth. Hence, to ensure that the wear measurements are accurate, the displacement results for wear are compared with the weight loss of the specimens and almost linear relationship is observed between the two for the range of test parameters considered in the present study.

3.6 Coating characterization

Characterization of the coating is necessary in order to ensure the proper development of the coating. Scanning electron microscopy (SEM) (JEOL, JSM 6360 and FEI Quanta 200) is performed on as deposited as well as heat treated coating surface. SEM is also performed on the worn out surface in order to get an idea about the wear mechanism. The composition of the coating is obtained with the help of energy dispersive X-ray analysis (EDAX Corporation). X-ray diffraction (XRD) analysis (Rigaku, Ultima III) is performed in order to identify the different phases before and after heat treatment.

4. EXPERIMENTAL RESULTS AND DISCUSSION

4.1 Analysis of signal to noise ratio

Taguchi method aims to keep the variance in the output response to the minimum, even if noise inputs are present. Hence, Taguchi method needs to take into account the variability within a trial condition. The variability can be easily captured if S/N ratio is used to convert the experimental results into a value for the evaluation characteristic in the optimum parameter analysis, instead of the mean. The idea is to maximize the S/N ratio, thereby minimizing the effect of random noise factors, which have a significant impact on the process performance. In the present work S/N ratio analysis is done with depth of wear as the performance index and all the calculations are conducted in Minitab [26]. As wear is to be minimized, S/N ratio is calculated using LB (Lower the Better) criterion and is given by:

$$S/N = -10\log\left(\sum y^2/n\right) \qquad (1)$$

where y is the observed data and n is the number of observations.

The COF values obtained from experimentation along with their S/N ratios is shown in Table 4. As it is known that the columns of the OA are orthogonal to each other, the average effect of each factor on the quality characteristic at different levels can be determined. The average of the S/N ratio for each level of the factors of L, S, and T are given in Table 5. The delta value was calculated by subtracting the largest value from the lowest from among the values in each column. Very simply, a design factor with a large difference in the signal noise ratio from one factor setting to another indicates that the factor or design parameter is a significant contributor to the achievement of the performance characteristic. When there is little difference in the signal to noise ratio from one factor setting to another, this indicates that the factor is insignificant with respect to the performance characteristic. It is found from Table 5 that load (L) possesses the highest delta value and hence has the greatest influence over the wear performance of electroless Ni-B coatings. The main effect and interaction effect plots are illustrated in Fig. 4 and Fig. 5 respectively. The main effects plot gives the optimal combination of testing parameters for minimum wear.

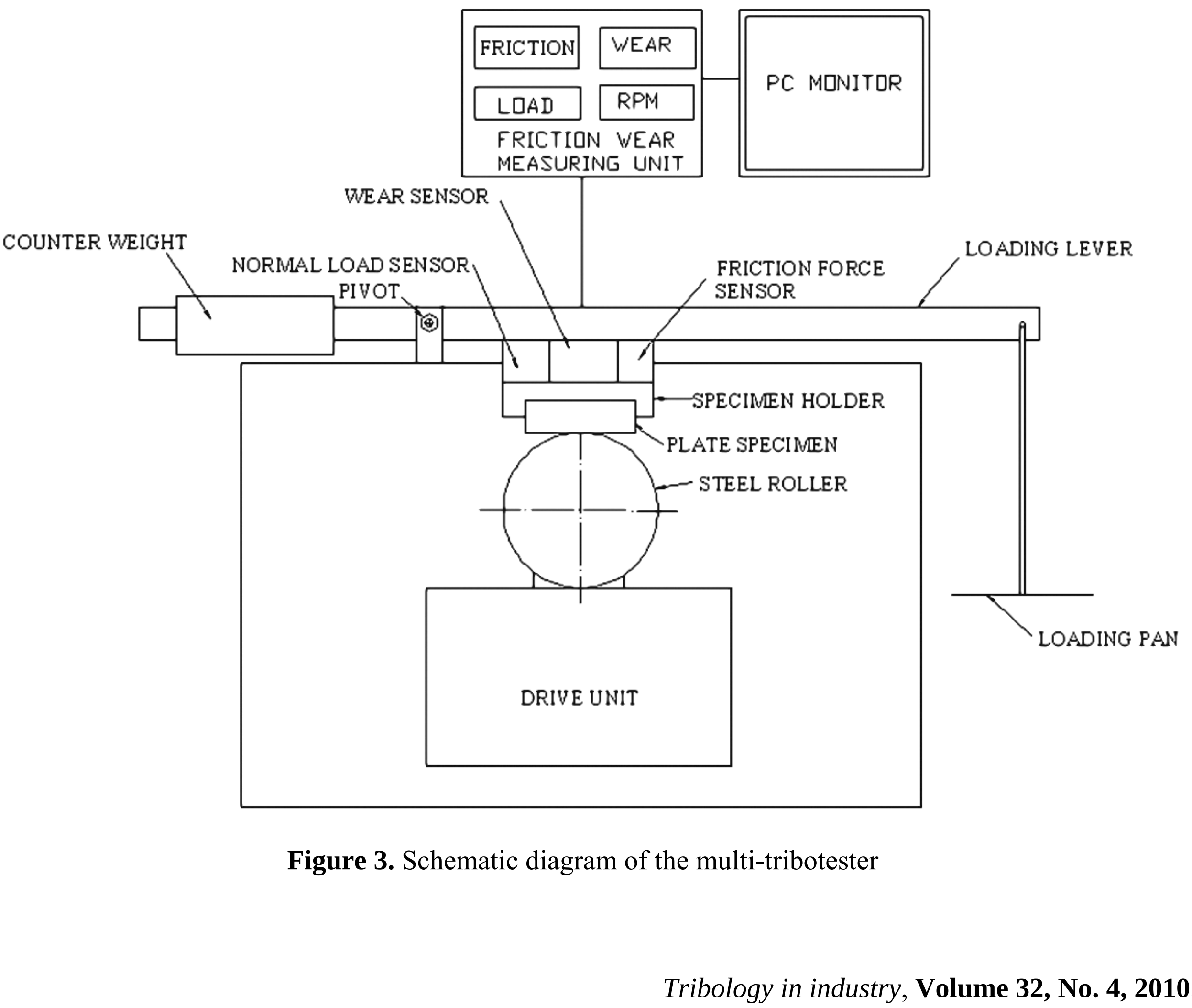


Figure 3. Schematic diagram of the multi-tribotester

Table 4. Experimental results along with S/N ratio

Exp No.	Wear (μm)	S/N Ratio
1	5.752	-15.1966
2	10.112	-20.0963
3	17.998	-25.1044
4	8.305	-18.3868
5	13.912	-22.8677
6	15.410	-23.7560
7	5.978	-15.5311
8	11.935	-21.5364
9	15.638	-23.8836
10	6.636	-16.4381
11	13.431	-22.5621
12	16.982	-24.5997
13	8.012	-18.0748
14	10.998	-20.8268
15	14.002	-22.9238
16	12.803	-22.1462
17	15.731	-23.9351
18	17.141	-24.6807
19	18.475	-25.3316
20	24.893	-27.9215
21	27.361	-28.7426
22	16.61	-24.4073
23	20.789	-26.3566
24	24.151	-27.6587
25	14.992	-23.5171
26	19.054	-25.5997
27	23.886	-27.5628

Table 5. Response table of mean S/N ratio

Level	L	S	T
1	-20.71	-22.89	-19.89
2	-21.80	-22.81	-23.52
3	-26.34	-23.15	-25.43
Delta	5.64	0.35	5.54
Rank	1	3	2

Total mean S/N ratio for wear = -22.9498 dB

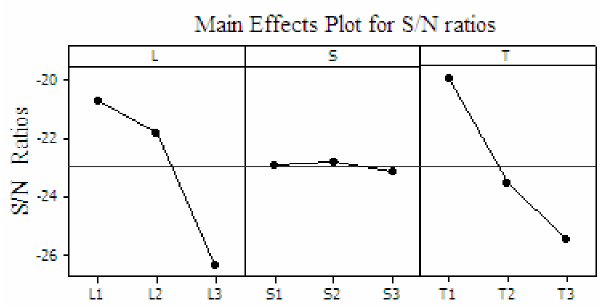
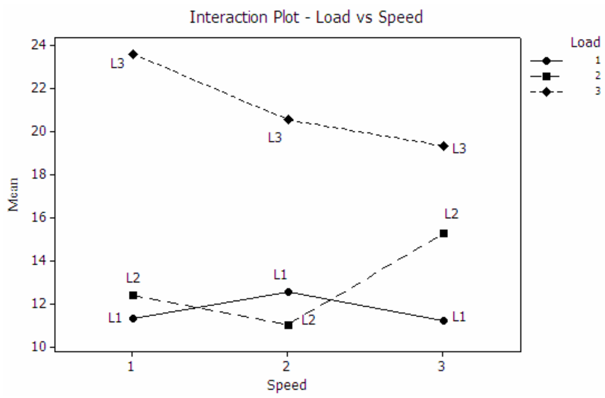
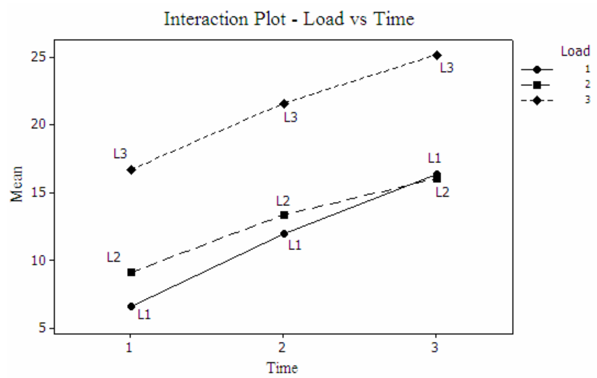


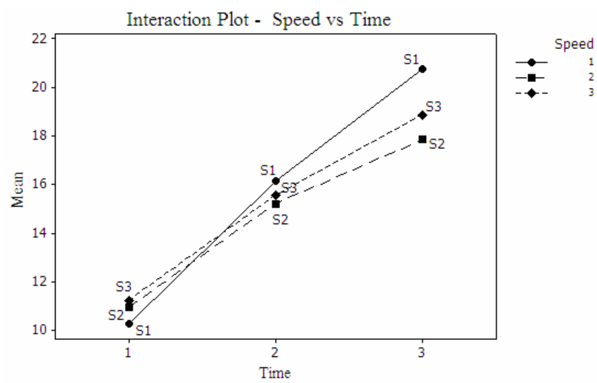
Figure 4. Main effects plot for signal to noise ratio



(a)



(b)



(c)

Figure 5. Interaction effects plot for mean wear (a) L versus S, (b) L versus T and (c) S versus T

Since Taguchi method obtains the optimal level combination by choosing those levels for which S/N ratio is the highest, the optimal combination of parameters is found to be L1S2T1, i.e. lower-level of load, middle level of speed and lower level of time. Moreover, the main effect plot also gives a rough idea about the relative significance of the parameters on the system response. This is determined by the slope of the main effect plot for each parameter. The plot having higher inclination will have higher influence. From Fig. 4, it can be observed that both the factors L (load) and T (time) are very much significant while factor S (speed) is almost horizontal and hence insignificant.

In case of interaction plots non-parallelism of the plots are observed. Non-parallel lines are indicative of the presence of interaction while intersecting lines are indicative of the presence of strong interaction. From the interaction plots (Fig. 5), it is evident that strong interaction exists between factors L and S (L×S) while moderate interaction exists between the rest of the factors (L×T and S×T) as far as the wear performance of electroless Ni-B is concerned.

4.2 Analysis of Variance (ANOVA)

ANOVA is a statistical technique to find out the significance of individual process parameters and their interactions on the system response under consideration. In the present study, ANOVA is applied with an objective to evaluate the significance of testing parameters and their interactions on the wear performance of electroless Ni-B coating. If some testing parameters do not have considerable impact on wear, they can be kept within a suitable range for the test and can be excluded in building future prediction and optimization models. The percentage contribution of variance can also be calculated through ANOVA. In the present study, ANOVA is performed using S/N ratio as the response and the results are shown in Table 6. The ANOVA table also consists of the F-values and the percentage contributions. By comparing the evaluated F values with the tabulated ones, the significance of the factors and their interactions can be readily understood. If the obtained F-value of a parameter or interaction is greater than the tabulated one, then that particular parameter or interaction has a significant influence over the process response. From Table 6, it can be seen that parameter L, i.e. load has got the most significant influence on wear at the confidence level of 99% within the specific test range. Parameter T, i.e. time is also very much significant at the same confidence level. But parameter S (speed) has almost no significance on the wear performance. So in future studies, speed can be kept at a constant level while other parameters could be included in the analysis to observe their effect on the wear performance of electroless Ni-B coatings. Among the interactions, the interaction between load and speed (L×S) and that between load and time (L×T) are significant at confidence levels of 95% and 90%.

Table 6. Results of ANOVA

Source	DF	SS	MS	F	% contribution
L	2	160.92	80.458	62.11[b]	44.83
S	2	0.597	0.299	0.23	0.17
T	2	142.67	71.333	55.07[b]	39.75
L×S	4	23.45	5.863	4.53[b]	6.53
L×T	4	14.83	3.707	2.86[b]	4.13
S×T	4	6.095	1.524	1.18	1.7
Error	8	10.36	1.295		
Total	26	358.92			

[b] - significant parameters and interactions
($F_{0.01,2,8} = 8.65$; $F_{0.05,4,8} = 3.84$; $F_{0.1,4,8} = 2.81$)

4.3 Confirmation test

The final step of the DOE is the confirmation test, which verifies if the optimum conditions suggested by the matrix experiment do indeed give the improvement projected. The verification experiment is performed by conducting a test with optimal settings of the factors and levels previously evaluated. The predicted value of the S/N ratio at the optimum level $\hat{\eta}$ is calculated as:

$$\hat{\eta} = \eta_m + \sum_{i=1}^{o} \left(\overline{\eta}_i - \eta_m\right) \qquad (2)$$

where η_m is the total mean S/N ratio, $\overline{\eta}_i$ is the mean S/N ratio at the optimal level, and o is the number of main design parameters that significantly affect the wear performance of electroless Ni-B coating. Table 7 shows the comparison of the predicted S/N ratio with the actual (experimental) S/N ratio using the optimal parameters and there seems to be quite a good agreement between the two. Moreover the improvement of S/N ratio at the optimal condition compared to the initial condition is also shown in the present table. The increase of the S/N ratio from the initial testing condition to the optimal testing condition is found to be 2.44 dB. This means that Ni-B coatings tested at optimal condition experienced about 25% reduction in wear compared to the ones tested at the initial condition. In other words, the experimental results confirm the prior design and analysis for optimizing the tribological test parameters is suitably applied for the present case.

Table 7. Results of the confirmation experiment

	Initial parameters	Optimal Parameters	Experi-ment
Level	L2S2T2	L1S2T1	L1S2T1
Wear	10.998		8.305
S/N ratio (dB)	-20.8268	-17.649	-18.3868

Improvement of S/N ratio = 2.44 dB

4.4 Microstructure study

Energy dispersive X-ray analysis is performed with one of the newer EDX detectors that do not contain any Beryllium window in order to detect light elements like boron. The Beryllium window if present absorbs all the soft X-rays thereby precluding the detection of lighter elements. The EDX plot is shown in Fig. 6 and boron content in terms of weight percentages is found to be about 5.72 while the remaining is mostly nickel.

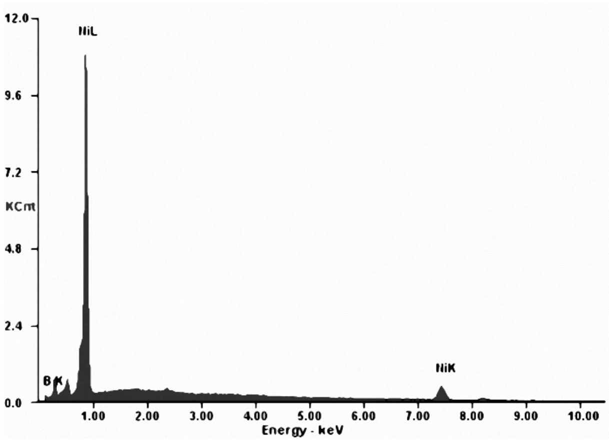
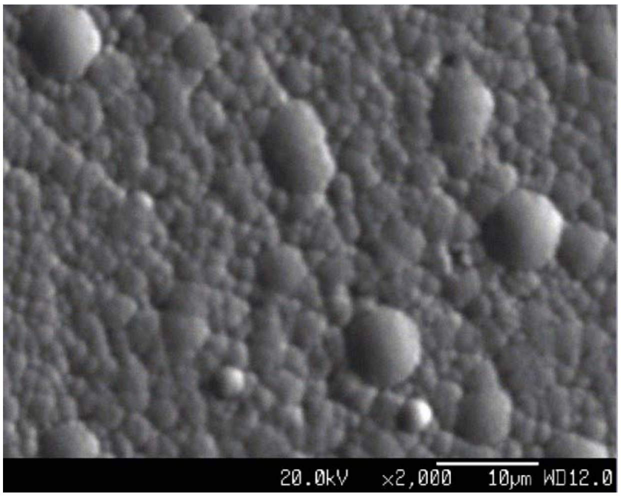
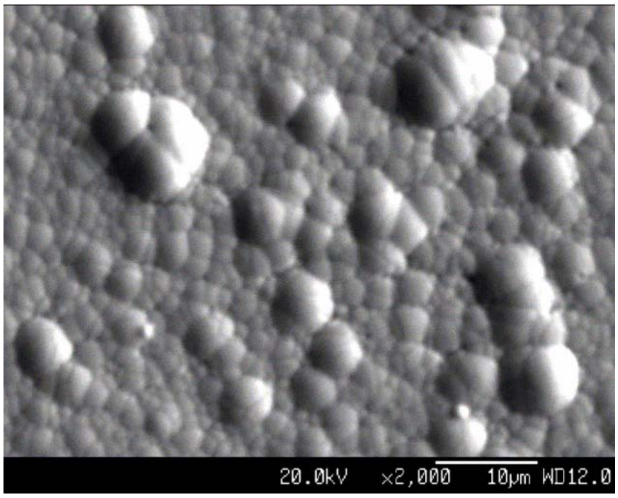


Figure 6. EDX spectra of Ni-B coating

Surface morphology study of the coatings is done by SEM in order to interpret the effect of heat treatment on the microstructure of the coatings for some of the samples at random and they show similar qualitative changes in microstructure. The SEM micrographs of the coated samples in as deposited and under heat treated conditions (at 350ºC) are shown in Fig. 7. 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 [8]. The surface of the coating appears dense and light grey in colour with low porosity. Also it can be noted by carefully observing the pictures that the Ni-B nodules are almost flat and uniformly dispersed in as deposited condition. But when heat treated, the 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 structure which becomes crystalline with heat treatment. This is further supported by the XRD patterns of Ni-B deposits in as deposited and heat treated condition (Fig. 8). The XRD patterns in as deposited condition is a collection of micro-crystalline peaks. But with heat treatment at 350°C for one hour, broad peaks of Ni_2B and Ni_3B are produced.

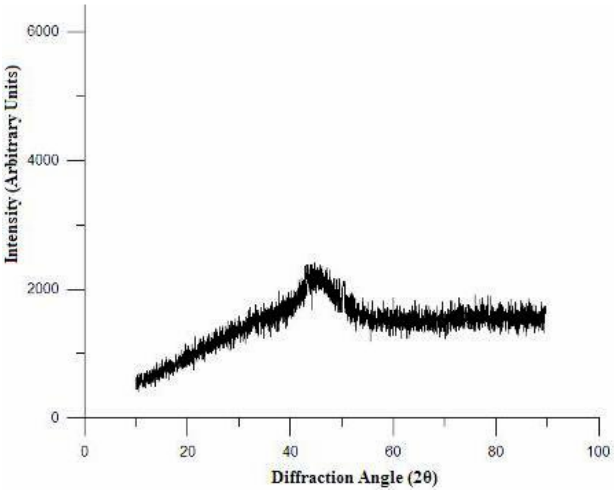


(a)

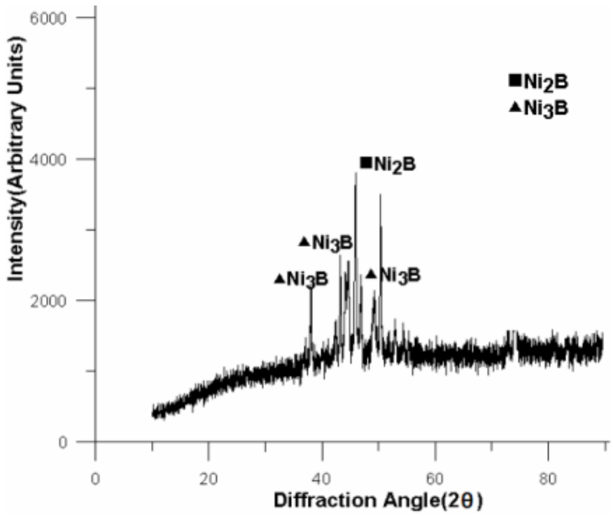


(b)

Figure 7. SEM pictures of the coated surface (a) as deposited (b) heat treated (350ºC, 1 hr)



(a)



(b)

Figure 8. XRD patterns of electroless Ni–B deposit (a) as-deposited and (b) heat treated at 350ºC

4.5 Wear mechanism

SEM micrograph of the worn surface after wear testing is shown in Fig. 9. It is clear from the figure that load is taken by some of the nodules while the others remain almost undeformed. The presence of longitudinal grooves along the sliding direction with high degree of plasticity can be clearly observed. This is indicative of the occurrence of micro-cutting and micro-ploughing effect and characterized as ductile failure. Almost no pits or prows are observed on the worn surface. Hence it can be concluded that the abrasive wear is the predominant phenomenon. The same trend is observed for other combinations of deposition parameters within the experimental regime considered in this study.

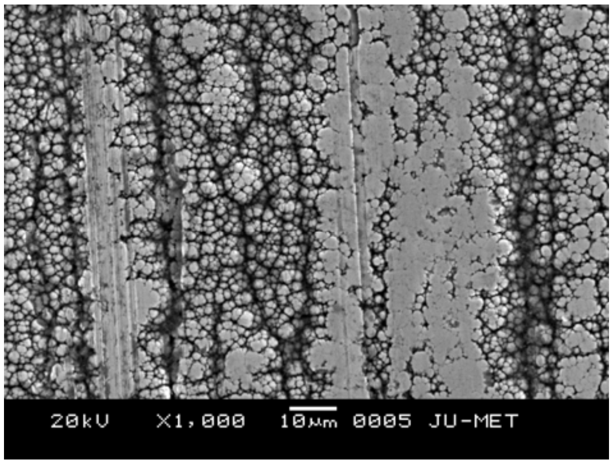


Figure 9. SEM picture of coating surface after friction testing

5. CONCLUSION

Taguchi orthogonal array design is suitably applied to minimize the wear of electroless Ni-B coatings by optimizing three tribological testing parameters viz. load (L), speed (S) and time (T). The optimum testing condition (L1S2T1) obtained from the analysis yields about 25% reduction in wear compared to the initial condition (L2S2T2). The ANOVA analysis reveals that load and time have a great influence over the wear performance of the coating. Moreover the analysis shows that the interaction between load and speed (L×S) and that between load and time (L×T) are also significant in controlling the wear performance. The SEM micrographs revealed that the coating possesses a cauliflower like structure with no obvious surface damage and is of low porosity. The coating also appears to be dense and light grey in colour. The XRD plots showed that the electroless Ni-B coating is a mixture of amorphous and crystalline phase in as deposited condition. But with heat treatment, the coating turns crystalline. This is ascertained by the presence of Ni_2B and Ni_3B peaks in the XRD plot of Ni-B coating heat treated at 350°C. The worn surface primarily shows longitudinal grooves along the sliding direction, indicating that the wear mechanism is mainly abrasive in nature.

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