Hindawi Publishing Corporation
Indian Journal of Materials Science
Volume 2015, Article ID 104187, 10 pages
http://dx.doi.org/10.1155/2015/104187

Research Article

Improvement in Tribological Performance of Ni-P-TiO_2 Composite Coatings Using Taguchi Technique with Grey Relational Analysis

Prasanna Gadhari and Prasanta Sahoo

Department of Mechanical Engineering, Jadavpur University, Kolkata 700032, India

Correspondence should be addressed to Prasanta Sahoo; psjume@gmail.com

Received 12 May 2015; Revised 30 June 2015; Accepted 2 July 2015

Academic Editor: Pradeep Lancy Menezes

Tribological performance of Ni-P-TiO_2 composite coatings is improved by varying the tribological test parameters such as normal load, wear track diameter, and duration of test aiming at minimum wear and friction of the coating. Taguchi technique with grey relational analysis is employed for optimization of multiresponse problem using L_{27} orthogonal array (OA). Analysis of variance (ANOVA) is used to find out the significant effect of test parameters and their interactions on friction and wear behavior of the coating. ANOVA results reveal that normal load and time (test duration) have the most significant effect in controlling wear and friction of the coating. Interaction between normal load and wear track diameter has some significant effect. Scanning electron microscopy of worn surface shows abrasive wear to be predominant. The surface morphology, composition, and phase structure analysis are done with the help of scanning electron microscopy (SEM), energy dispersive X-ray (EDX) analysis, and X-ray diffraction (XRD) analysis, respectively.

1. Introduction

Machine components having mating surfaces are prone to failure due to wear and corrosion. Surface coatings substantially improve the performance of such mating surfaces. Electroless nickel (EN) coatings provide cost effective way to achieve excellent tribological properties. It is an autocatalytic deposition of a nickel-phosphorus alloy from an aqueous solution on the substrate surface without application of an electric current. Electroless composite coatings are widely used in machinery (mechanical) and computer memory disc and automotive, electronic, oil and chemical, food, textile, and printing industries. Electroless nickel coating containing fine inert second phase particles deposited within the Ni-P matrix is known as electroless Ni-P composite coating. The soft composite coatings are produced by embedding soft particles (MoS_2, PTFE, and WS_2) and hard composite coatings are produced by embedding hard particles (TiO_2, Al_2O_3, B_4C, SiC, and diamond) [1]. Effective embedding of composite particles in the Ni-P layer depends on bath composition, bath reactivity, compatibility of the composite particles with the metallic matrix, plating rate, and particle size distribution [2]. The significant improvement in the properties of EN composite coatings depends on stable dispersion of particles (microsize/nanosize). Second phase particles may be agglomerated due to nonuniform dispersion in electroless bath. Agglomeration of particles in the electroless bath results in formation of severe defects in the coatings [3]. Incorporation of second phase particles in the Ni-P matrix depends on two important factors, namely, impingement of second phase particles on the coated surface and time available to hold the particle on the coated surface [4]. Hardness and wear resistance of EN composite coatings are correlated. Hardness and tribological properties of the composite coatings are significantly improved after heat treatment at 400°C/hour in muffle furnace followed by cooling them down to room temperature in the same furnace [5]. Uniform distribution of composite particles in Ni-P matrix layer plays an important role in improvement of microhardness and wear resistance of the composite coatings. It is possible to improve tribological

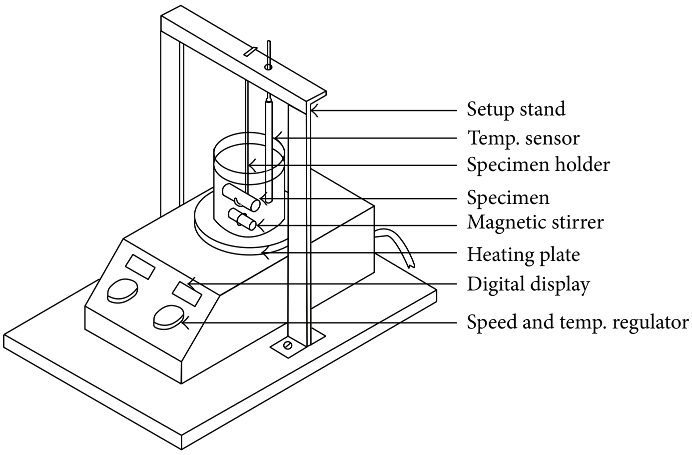


Figure 1: Experimental setup for electroless Ni-P-TiO_2 composite coating.

and mechanical properties using a special technique, sol-gel technique, in which second phase particles are not agglomerated in the electroless bath [6].

Wear is in general measured either in terms of weight loss of coating or wear depth of wear track produced on the coated surface. X-ray diffraction method is used instead of weight loss method for measurement of wear resistance [7]. Hamid and Abou Elkhair [8] have compared the wear resistance of aluminum alloy substrate, Ni-P, Ni-P-TiO_2, Ni-P-ZrO_2, and Ni-P-Al_2O_3 composite coatings in as-deposited and heat treated (400°C) conditions and found excellent wear resistance and microhardness of Al_2O_3 composite coating compared to the other coatings and alloy substrate. After heat treatment (400°C), Al_2O_3 composite coatings exhibited excellent wear resistance compared to Ni-P coatings due to formation of hard Ni_3P alloy phase [9]. The Ni-P-TiO_2 composite coatings have excellent wear resistance (lower width of wear track) compared to conventional Ni-P coating [6]. The Ni-P-nano-Al_2O_3 composite coating has higher hardness and excellent wear resistance to adhesive wear compared to Ni-P and Ni-P-Al_2O_3 (micro) composite coatings [10]. From extensive literature review it is found that wear resistance of the base material significantly improves in the presence of hard and soft second phase particles in the Ni-P layer [11–13].

The present study deals with the tribological properties (friction and wear) of the Ni-P-TiO_2 composite coatings. It is essential to reduce the wear rate and friction coefficient of the composite coating. From the literature review it is revealed that hardness, wear rate, and friction coefficient depend on heat treatment and the amount of hard particles present in the coating. Hence the present study deals with optimization of tribological parameters used in tribotesting to reduce wear rate and friction of the coating. Taguchi method combined with grey relational analysis is employed to optimize the process parameters in order to identify the combination of parameters that yields minimum wear and friction coefficient of Ni-P-TiO_2 composite coating. Analysis of variance (ANOVA) is used to observe the level of significance of the factors and their interactions. The surface morphology and composition of Ni-P-TiO_2 coatings are studied with the help of scanning electron microscopy (SEM), energy dispersed X-ray (EDX) analysis, and X-ray diffraction (XRD) analysis.

2. Materials and Methods

2.1. Substrate Preparation and Experimental Setup for Ni-P-TiO_2 Composite Coating. As per the requirement of tribotest setup, pin type samples (30 mm length and 6 mm diameter) are used for coating deposition. In the present work mild steel (AISI 1040) is used as substrate material. Effective deposition of coating on substrate depends on the preparation of the substrate. Therefore the substrates are prepared carefully. Before coating deposition process, substrate is subjected to pickling treatment using dilute hydrochloric acid solution (50% HCl (36% pure) + 50% deionized water) for few seconds to remove layer formed due to rust and other oxides. After that the substrate is rinsed with deionized water and methanol. The substrate is activated in warm palladium solution to boost the coating deposition rate. After that, the activated substrate is immediately dipped into a hot electroless bath, maintained at 85°C. The coating deposition process is carried out for three hours. For each specimen the constant deposition time is maintained to obtain uniform thickness of the coating. The thickness of the coating is found in the range of 25 to 29 microns. After completion of the coating deposition process, the samples are cleaned with deionized water and then wrapped in soft paper to protect them from environmental changes.

Figure 1 shows the experimental setup for deposition of electroless Ni-P-TiO_2 composite coating. Setup consists of heater cum magnetic stirrer (IKA RCT basic) with temperature range from 0 to 310°C and stirrer speed ranges from 0 to 1500 rpm. A rigid stand is used to hold and support the substrate and glass coated temperature sensor. Glass beaker (250 mL) containing the electroless solution (200 mL) is kept on the heating plate for coating deposition. Magnetic stirrer is used to maintain the suspension of particles in the bath and to avoid agglomeration of TiO_2 particles.

Table 1: Chemical composition and working conditions for electroless bath.

Bath chemical composition	Quantity	Working conditions	
$NiSO_4 \cdot 6H_2O$ (nickel sulphate)	45 g/L	pH	4.5–5.0
$NaH_2PO_2 \cdot 2H_2O$ (sodium hypophosphite)	20 g/L	Bath temperature	85 ± 2°C
TiO_2 particles	5 g/L	Bath volume	250 mL
$C_6H_5Na_3O_7 \cdot 2H_2O$ (trisodium citrate)	15 g/L	Stirrer speed	300 rpm
CH_3COONa (sodium acetate)	5 g/L	Deposition time	3 hours
$C_{12}H_{25}NaO_4S$ (sodium dodecyl sulphate)	0.2 g/L	Annealing temperature	400°C

2.2. Electroless Bath Preparation for Ni-P-TiO$_2$ Composite Coating. The electroless coating is developed using different types of chemicals and working conditions (process parameters) in the electroless bath. These parameters are responsible for varying the performance characteristics of the electroless coatings. Hence, it is very essential to select the appropriate process parameters along with their quantity. The bath composition and operating conditions of the electroless Ni-P-TiO$_2$ composite coatings are shown in Table 1.

For better suspension and to avoid agglomeration of TiO$_2$ particles, the proper amount of surfactant (sodium dodecyl sulphate (SDS)) is added to the electroless bath. Approximately 50 mL of electroless Ni-P solution contained with specified amount of TiO$_2$ particles and SDS underwent thorough mixing using magnetic stirrer (Remi make 2 MLH) to get better suspension of TiO$_2$ particles in the electroless bath. At first Ni-P coating is deposited for one hour to prevent the porosity of the coating, and then the slurry of TiO$_2$ particles, SDS, and electroless bath (50 mL) is introduced in the same bath for the subsequent two hours to deposit TiO$_2$ composite coating.

2.3. Function of Coating Process Parameters. In the electroless bath, nickel sulphate is used as nickel source (it supplies nickel ions in the solution) and sodium hypophosphite is used as reducing agent which reduces the nickel ions from their positive valence state to zero valence state. At 85°C bath temperature, the chemical reaction between nickel source and reducing agent is quite rapid and vast, which may result in decomposition of the electroless bath. To avoid bath decomposition due to rapid and vast chemical reaction, complexing agents are used to stabilize the electroless bath. Sometimes bath has decomposed even though complexing agents are present in the electroless bath; in such situation, stabilizers play an important role to avoid the decomposition of bath. Surfactants are used to increase the wettability and surface charge of composite particles [14, 15].

2.4. Measurement of Wear and Friction. The tribological study of heat treated electroless Ni-P-TiO$_2$ composite coating is performed using a tribotester apparatus (TR-20LE-CHM-400, DUCOM). The tests are carried out using pin-on-disc geometry in dry (unlubricated) condition. The ambient temperature is about 32°C and relative humidity is approximately about 80%. During tribological test, normal load is varied from 50 N to 100 N, wear track diameter is varied from 80 to 120 mm, and duration of trial is varied from 300 seconds to 900 seconds and disc is rotated at 100 rpm. Figure 2 shows the schematic diagram for pin-on-disc tribotester. The Ni-P-TiO$_2$ composite coated pin sample is held stationarily with the help of specimen holder and is ready to slide against a rotating wear disc having hardness of 62 HRc. The rotational speed of wear disc and duration of trial are controlled with the help of a computer attached to the tribotester. The load cell measures the friction between coated pin sample and rotating disc and LVDT measures the wear between coated specimen and disc.

Wear displacement is the sum of the wear on specimen surface and that on the counter face surface. The hardness of the coatings is found around 50 HRc, which is lower than the wear disc material. The wear disc material encounters negligible wear compared to the coated specimen. Hence, the measured displacement is taken as a representative of the actual wear depth encountered by the coating surface. In general wear is measured in terms of wear volume or mass loss. In the present study, wear is expressed in terms of displacement or wear depth (μm). Hence, to ensure accurate wear measurement, 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.

2.5. Microstructure Study and Characterization of Ni-P-TiO$_2$ Composite Coating. Energy dispersive X-ray analysis (EDAX Corporation) is used to find out the presence of nickel, phosphorus, titanium, and oxygen in composite coating in terms of weight percentages. Scanning electron microscopy (JEOL, JSM-6360) is used to observe the surface morphology of the composite coating before and after heat treatment (400°C). X-ray diffraction analyzer (Rigaku, Ultima III) is used to find out the phase composition of as-deposited and heat treated composite coatings.

2.6. Taguchi Method with Grey Relational Analysis and Experiment Planning. The design factors or input parameters are the experimental parameters which are varied within a specific range to obtain a desired result of the response variables (output parameters). The aim of the study is to obtain an optimum combination of design factors for the best possible value of response variables (output parameters). There are many factors which can be considered for controlling the tribological behavior of Ni-P-TiO$_2$ composite coatings. However, literature review shows that the normal load (A), wear track diameter (B), and duration/time of test (C) are the most widespread parameters amongst the researchers to

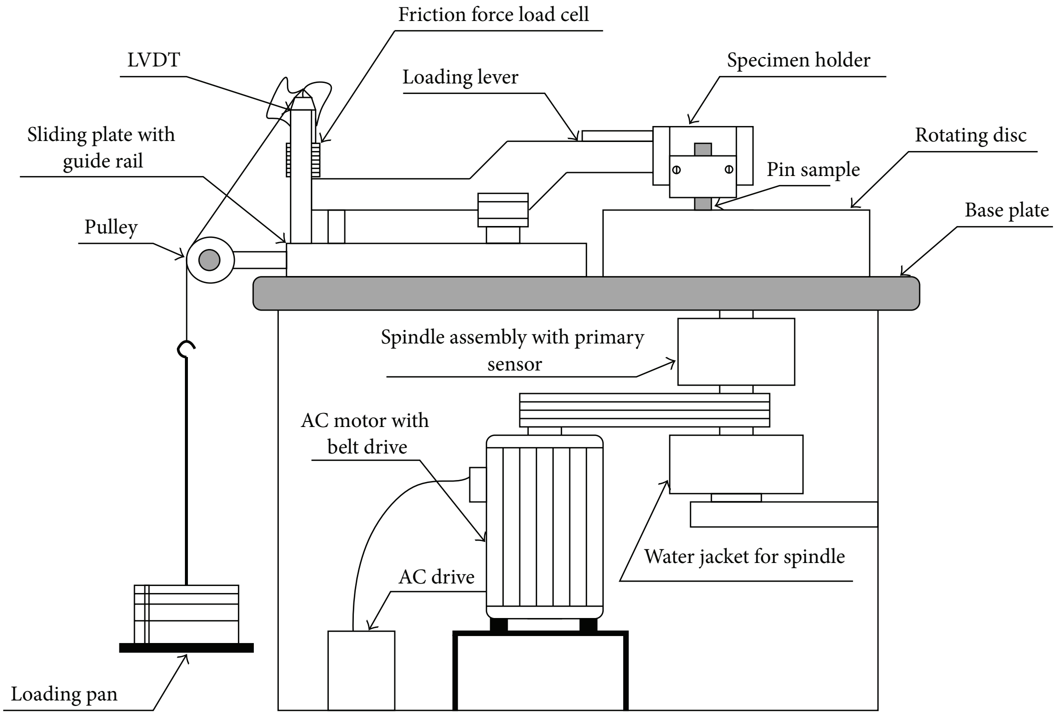


Figure 2: Experimental setup of pin-on-disc tribotester.

control the tribological performance of coatings. These three factors are considered as the main design factors along with their interactions. Table 2 shows the design factors along with their levels.

The present study tries to utilize Taguchi method [16] to find the best possible optimum combination of process parameters to achieve better tribological performance of Ni-P-TiO_2 composite coatings. On the basis of literature review [17–19], the most influential process parameters are selected to improve the tribological performance of the composite coating. The quality characteristics that need to be improved include minimization of wear depth and friction coefficient of the composite coating. Both of these tribological characteristics possess the-smaller-the-better property. The study considers three equally spaced levels for each design factor to find the impact of noise factors. According to the number of design factors and their levels and considering the effect of individual factors as well as the interactions, L_{27} orthogonal array is selected to proceed with the experiments. As friction and wear are two distinct responses, it may be possible that higher S/N ratio for one response corresponds to the lower S/N ratio for the other. Hence, such multiresponse problem can be solved by grey relational analysis [20]. It converts multiresponse (variable) problem into single response problem. It is used for overall evaluation of the S/N ratio to optimize the multiple response characteristics. The grey relational grade is treated as the overall response of the process instead of multiple responses of wear depth and friction coefficient.

Table 2: Design parameters and their levels.

Design factors	Designation	Unit	Levels		
			1	2	3
Normal load	*A*	N	50	75^*	100
Wear track diameter	*B*	mm	80	100^*	120
Duration of test	*C*	Seconds	300	600^*	900

*Initial condition.

3. Result and Discussion

3.1. Optimization of Triboparameters and Validation Test. The experimental values of wear depth and friction coefficient are given in Table 3. The present work deals with two responses, wear depth and friction coefficient, for optimization of tribological behavior of Ni-P-TiO_2 composite coatings. The particular sets of analyses are performed to convert the given multiple responses into a single performance index, called grey relational grade.

Experimental results of the linear normalization, that is, friction coefficient and wear depth, in the range of zero and one, are essential for generating the grey relational coefficient. For excellent tribological performance, the-lower-the-better criterion is used for normalization of the multiresponse parameters. Larger normalized results correspond to excellent performance and the best normalized result should be equal to one.

Table 3: Experimental values of friction coefficient and wear depth.

Sr. number	Friction coefficient	Wear depth (μm)
1	0.147	1.34
2	0.1672	3.53
3	0.186	5.25
4	0.1626	4.89
5	0.1786	7.87
6	0.2394	9.53
7	0.2494	6.37
8	0.2664	10.34
9	0.3556	13.1
10	0.134	2.53
11	0.1589	4.03
12	0.1936	6.43
13	0.1377	4.25
14	0.1618	9.92
15	0.2148	12.28
16	0.172	10.64
17	0.256	13.42
18	0.3272	16.35
19	0.2472	14.88
20	0.26	17.72
21	0.298	20.64
22	0.1349	15.56
23	0.1812	21.83
24	0.2342	24.71
25	0.1534	12.57
26	0.2686	17.54
27	0.291	23.63

Table 4: Grey relational grade and its order.

Sr. number	Grey relational grade	Order
1	0.947496	2
2	0.805803	5
3	0.714934	8
4	0.780909	6
5	0.677251	11
6	0.550206	15
7	0.594452	14
8	0.510247	18
9	0.415867	24
10	0.953786	1
11	0.814688	4
12	0.673404	12
13	0.884151	3
14	0.688016	9
15	0.547376	16
16	0.650725	13
17	0.483817	19
18	0.401098	25
19	0.478937	20
20	0.444379	21
21	0.39016	26
22	0.721507	7
23	0.532218	17
24	0.429226	23
25	0.680463	10
26	0.435275	22
27	0.378835	27

Experimental results of the linear normalization, that is, friction coefficient and wear depth, in the range of zero and one, are essential for generating the grey relational coefficient. For excellent tribological performance, both friction coefficient and wear depth are to be minimized. Hence the-lower-the-better criterion is used for normalization of the multiresponse parameters. The equation for the-lower-the-better criteria is given as follows:

$$x_i(k) = \frac{\max y_i(k) - y_i(k)}{\max y_i(k) - \min y_i(k)}, \qquad (1)$$

where $x_i(k)$ is the normalized value after grey relational generation, while $\min y_i(k)$ and $\max y_i(k)$ are the smallest and largest values of $y_i(k)$ for the k_{th} response, with $k = 1$ for friction coefficient and $k = 2$ for wear depth. Larger normalized results correspond to excellent performance and the best normalized result should be equal to one. The normalized values and grey relational coefficients are omitted here for brevity. The grey relational coefficient is calculated from the normalized value and the equation for the grey relational coefficient is as follows:

$$\xi_i(k) = \frac{\Delta_{\min} + r\xi\Delta_{\max}}{\Delta_{oi}(k) + r\xi\Delta_{\max}}, \qquad (2)$$

where $\Delta_{oi} = \|x_o(k) - x_i(k)\|$ is the difference of the absolute value between $x_o(k)$ and $x_i(k)$. $\Delta_{\min}$ and $\Delta_{\max}$ are the minimum and maximum values of the absolute differences (Δ_{oi}) of all comparing sequences. "r" is the distinguishing coefficient which is used to adjust the difference of the relational coefficient in the range of 0 to 1. The distinguishing coefficient weakens the effect of $\Delta_{\max}$ when it gets too big, enlarging the different significance of the relational coefficient. The suggested value of the distinguishing coefficient is 0.5, due to the moderate distinguishing effects and good stability of outcomes. Therefore, r is adopted as 0.5 for further analysis in the present case. The overall multiple response characteristics evaluation is based on grey relational grade and is calculated as follows:

$$\gamma_i = \frac{1}{n}\sum_{i=1}^{n}\xi_i(k), \qquad (3)$$

where n is the number of process responses. The grey relational grades are considered in the optimization of multiresponse parameter design problem. The values of grey relational grade are shown in Table 4.

Taguchi method transforms the loss function into a concurrent statistic (S/N ratio), which combines both the mean level of the quality characteristics and variance around the

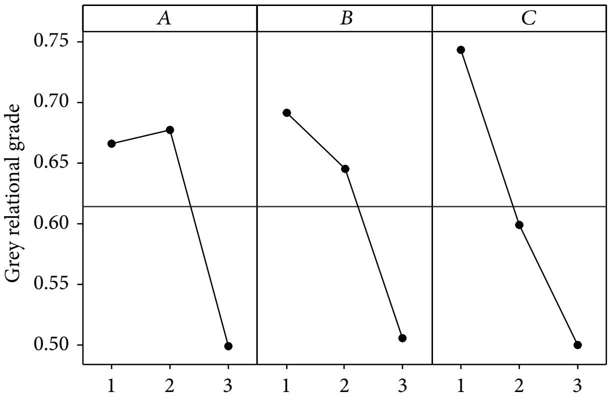


Figure 3: Main effects plot for grey relational grade.

Table 5: Mean response table for grey relational grade.

Level	A	B	C
1	0.666352	0.69151	0.743603
2	0.677451	0.645651	0.599077
3	0.499	0.505642	0.500123
Delta	0.178451	0.185868	0.24348
Rank	3	2	1

Total mean grey relational grade = 0.614268.

Table 6: Result of ANOVA for tribological behavior.

Source	DOF	SS	MS	F	% contribution
A	2	0.179924	0.089962	47.81202*	22.732
B	2	0.168757	0.084378	44.84453*	21.3218
C	2	0.269887	0.134943	71.71821*	34.09917
$A \times B$	4	0.144399	0.0361	19.18592*	18.24429
$A \times C$	4	0.006362	0.00159	0.84524	0.803756
$B \times C$	4	0.007095	0.001774	0.942677	0.896412
Error	8	0.015053	0.001882		1.901842
Total	26	0.791475			100.0000

*Significant at 99.9% confidence level ($F_{2,8,0.001} = 18.4937$ and $F_{4,8,0.001} = 14.3916$).

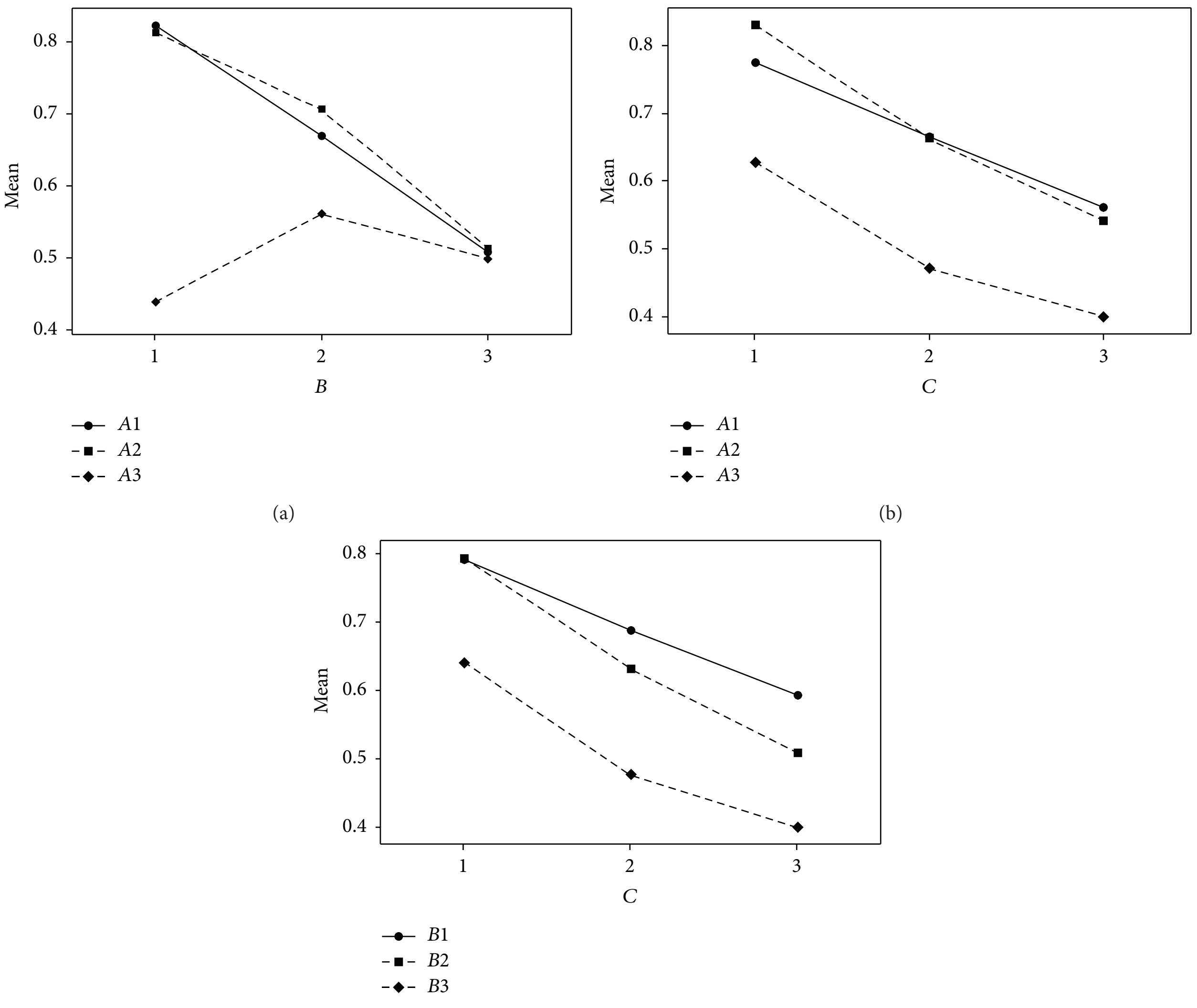
mean into a single metric. The S/N ratio consolidates several repetitions into one value. A larger S/N ratio represents a better quality characteristic. The S/N ratio is maximized to reduce the effect of random noise factors and to identify the significant effects of the process parameters. The S/N ratio for grey relational grade is calculated using the-higher-the-better criteria. The mean grey relational grade for three levels of the three factors is summarized in Table 5. All the calculations are performed with the help of Minitab software [21]. The response table shows the average of the selected characteristic for each level of the factors. The ranks shown in the table are based on delta statistics that compares the relative magnitude of effects. The parameter possessing higher delta value has greater influence over the response. From Table 5, it is confirmed that parameter *C*, that is, test duration (time), has the highest delta value. Figure 3 shows the main effects plot for grey relational grade and Figure 4 shows the interaction plots between the process parameters.

From Figure 3, it is confirmed that normal load (parameter *A*) has some significant effect while duration/time of test (parameter *C*) has the most significant effect and wear track diameter (parameter *B*) has less significant effect. The main effects plot gives the optimal combination of testing parameters for desired tribological performance. Better multiresponse characteristics are obtained at higher value of grey relational grade. The optimal combination of parameters was found as *A*2*B*1*C*1. Interaction between parameters *A*, *B*, and *C* is shown in Figure 4. From the plots, it is confirmed that almost all lines are intersecting with each other; that is, all factors have some amount of interaction between each other. From the figure, considerable interaction between parameters *B* and *C* and between parameters *A* and *C* is seen. Thus, from the present analysis, it is evident that strong interaction exists between factors *A* and *B*.

ANOVA is useful to investigate the effect and significance level of the process parameters. This technique gives various important conclusions based on the analysis of the experimental data. ANOVA separates the total variability of the response into contribution of each of the factors and the error. The sophisticated software Minitab is used to obtain the results through ANOVA based on grey relational grade. ANOVA results for tribological behavior of the composite coatings are shown in Table 6. These calculations are based on the *F*-ratio or variance ratio. *F*-ratio is used to measure the significance of the parameters under investigation with respect to variations of all the terms included in the error term at the desired significance level. If the calculated value is higher than tabulated value, this means that factor is significant at the desired level. The ANOVA table shows the percentage contribution of each parameter. From the table it is confirmed that parameter *C* (test duration or time) has the most significant effect on the tribological performance at the confidence level of 99.99% within the specific test range. The normal load (*A*) and wear track diameter (*B*) are also significant at 99.99% confidence level. Among the interactions, the interaction of parameters between *A* and *B* has the significant contribution at confidence level of 99.99%. The percentage contributions of the factors and interactions are calculated to identify the influence of the process parameters. From ANOVA table, it is clear that parameter *C* has the largest contribution (34.099%) followed by parameter *A* (22.732%). Among interactions, interaction $A \times B$ has the highest contribution (18.244%).

In the final step, confirmation test is conducted on the basis of optimum combination of parameters with their respective levels. Table 7 shows the values of grey relational grade at initial condition and optimum condition (predicted and experimental). The increase in the grey relational grade from initial condition (*A*2*B*2*C*2) to the optimal condition (*A*2*B*1*C*1) is about 0.26577. This is more than 38.62% of the mean grey relational grade; it means significant improvement in tribological performance of composite coating.

SEM of the worn surface after wear test is shown in Figure 5. From the figure, it is confirmed that load is taken by



(a)

(b)

(c)

Figure 4: Interaction plots (a) between *A* and *B*, (b) between *A* and *C*, and (c) between *B* and *C*.

Table 7: Result of validation test.

	Initial condition	Optimum condition	
		Prediction	Experimentation
Level	*A2B2C2*	*A2B1C1*	*A2B1C1*
Friction coefficient	0.1618		0.134
Wear depth (μm)	9.92		2.53
Grade	0.688016	0.872929	0.953786

Improvement of grey relational grade = 0.26577.

some of the nodules while there is no change in other nodules (almost undeformed). The presence of longitudinal grooves along the sliding direction with high degree of plasticity is clearly observed. This indicates the occurrence of microcutting and ploughing effect (characterized as ductile failure). Almost no pits are observed on the worn surface. Hence, it is concluded that the abrasive wear is the predominant mechanism. The same trend is observed for other combinations of deposition parameters within the experimental regime considered in this study.

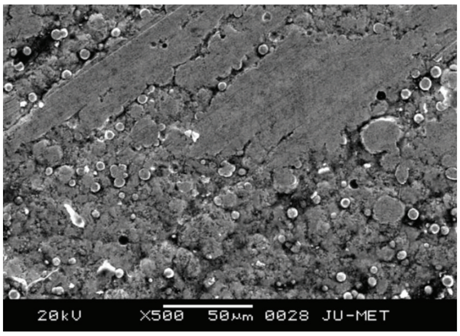
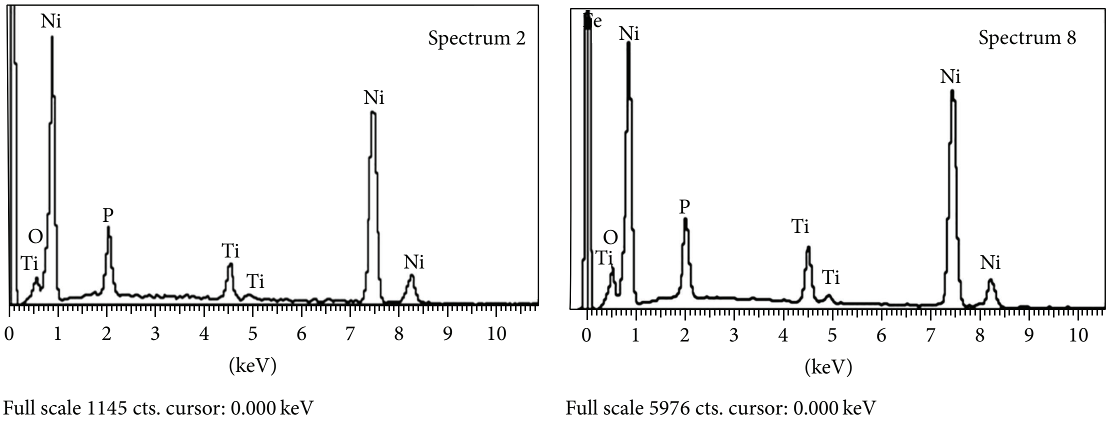


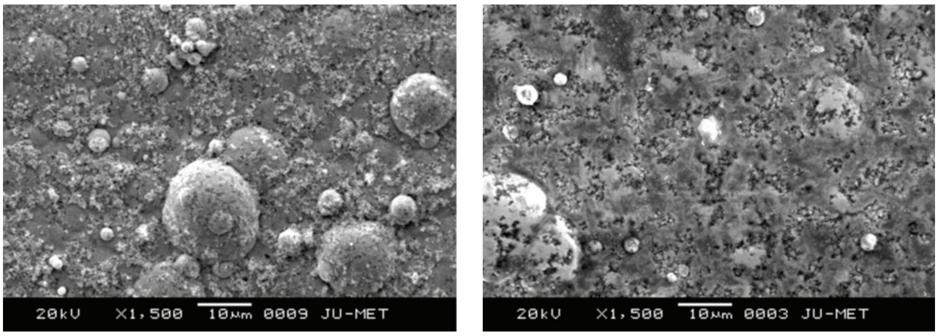
Figure 5: SEM image of worn surface of the Ni-P-TiO$_2$ composite coating.

3.2. Microstructure Analysis of Ni-P-TiO$_2$ Composite Coating. The characterization of the coating is essential to find out



(a) (b)

Figure 6: EDX of Ni-P-TiO_2 composite coating with (a) 5 g/L and (b) 10 g/L of TiO_2.



(a) (b)

Figure 7: SEM images of Ni-P-TiO_2 composite coatings: (a) as-deposited and (b) heat treated at 400°C.

the quantity of the coating elements. Energy dispersive X-ray (EDX) analysis is used to find out presence of various elements (nickel, phosphorus, titanium, and oxygen) in terms of weight percentage in the coating. Table 8 shows the EDX result of Ni-P-TiO_2 composite coatings at different concentration of titanium particles in the bath. Figure 6 shows the EDX plots for TiO_2 composite coatings, having different concentrations of TiO_2 particles (5 g/L and 10 g/L) in the electroless bath. From EDX plots and tabulated results, it is confirmed that the weight percentage of TiO_2 particles in the coating is increased (from 6.41 wt.% to 9.97 wt.%) with increase in concentration of TiO_2 particles in the electroless bath.

The SEM images of as-deposited and heat treated Ni-P-TiO_2 composite coatings are shown in Figure 7. The surface of the as-deposited specimen (Figure 7(a)) shows the typical nodular structure with incorporation of TiO_2 particles. Figure 7(b) shows the SEM micrographs of heat treated Ni-P-TiO_2 composite coatings at 400°C. From the figure, it is confirmed that the grain structure of the heat treated composite coatings changed due to the heat treatment. Also by careful observation, it is noticed that the grains are coarsened due to heat treatment at 400°C. At this temperature, crystallization of nickel and precipitation of phosphide (Ni_3P) occur, which results in increase in wear resistance.

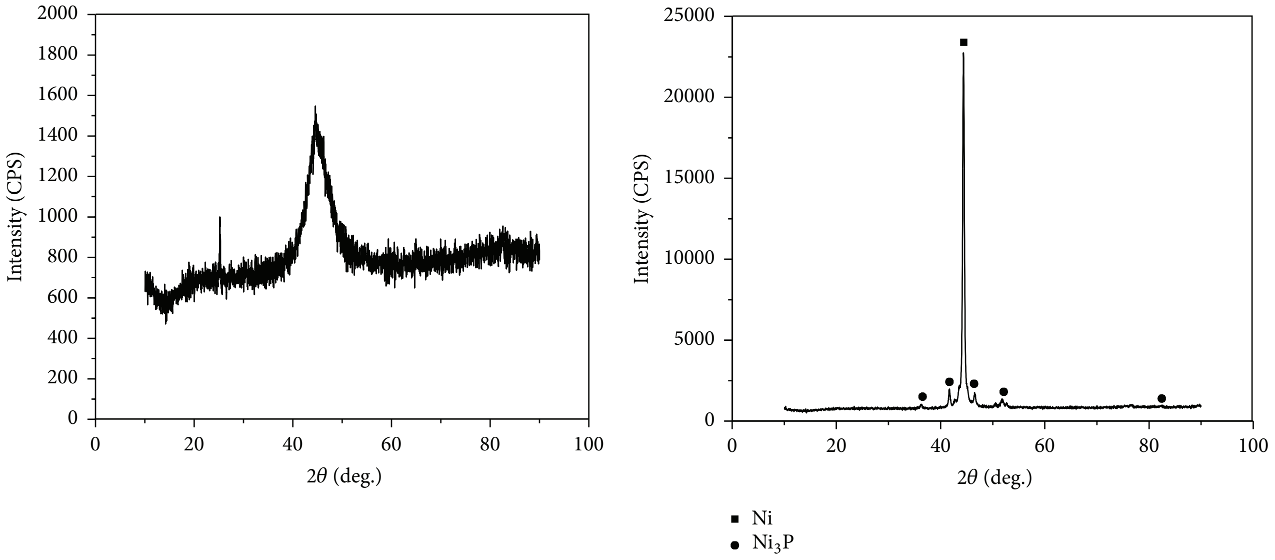
Figure 8 shows the X-ray diffraction plots for Ni-P-TiO_2 composite coatings in as-deposited and heat treated condition (400°C). Figure 8(a) shows XRD plot for as-deposited TiO_2 composite coating. From the plot, it is confirmed that the coating is almost amorphous in nature with single broad peak at diffraction angle of 44.24. Even though EDX results confirmed the presence of TiO_2 particles in the as-deposited coating, diffraction corresponding to TiO_2 particles is not traced in XRD analysis; it may be due to low quantity and highly dispersive distribution of the TiO_2 composite particles. Figure 8(b) shows the XRD plot for heat treated (at 400°C) TiO_2 composite coating. From the plot, it is confirmed that the amorphous phase is converted into crystalline phase. Due to heat treatment, peaks of Ni_3P (hard alloy phase) were seen at various diffraction angles. Ni_3P alloy phase is responsible for increase in hardness and wear resistance of TiO_2 composite coatings.

4. Conclusion

Ni-P-TiO_2 composite coatings are tested for tribological behavior using pin-on-disc tribotester. The design parameters are optimized in order to minimize friction and wear of the coatings. All design parameters have significant

Table 8: EDX results of Ni-P-TiO_2 composite coatings.

Figure number	Concentration of TiO_2 particles	% of Ni	% of P	% of O	% of Ti	Total
Figure 6(a)	5 g/L	80.72	8.13	4.74	6.41	100
Figure 6(b)	10 g/L	75.17	7.74	7.12	9.97	100



(a) (b)

Figure 8: XRD plots for Ni-P-TiO_2 composite coating in (a) as-deposited and (b) heat treated phase at 400°C.

influence on the friction and wear performance of the coating at a confidence level of 99.99%. The interaction between normal load and wear track diameter ($A \times B$) has significant influence on the tribological performance at a confidence level of 99.99%. Grey relational analysis is successfully employed in combination with Taguchi design of experiments to optimize multiple response problems. The optimal combination of tribological parameters was obtained as $A2B1C1$ (the lowest levels of wear track diameter and time and the middle level of normal load). EDX analysis confirms that the composite coating consists of nickel, phosphorus, and titanium particles. SEM micrographs reveal that the coating has a cauliflower-like structure without surface damage and low porosity. TiO_2 particles are uniformly distributed over the Ni-P coated layer. The coating also appears to be dense and light grey in color. From XRD plot, it is confirmed that the coating has amorphous structure in as-deposited condition and crystalline structure in heat treated condition (400°C). The pattern of the sliding tracks shows that abrasive failure is the predominant wear mechanism.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

References

[1] A. Sharma and A. K. Singh, "Electroless Ni–P and Ni–P–Al_2O_3 nano-composite coatings and their corrosion and wear resistance," *Journal of Materials Engineering and Performance*, vol. 22, no. 1, pp. 176–183, 2013.

[2] G. O. Mallory and J. B. Hadju, *Electroless Plating: Fundamentals and Applications*, AESF, Orlando, Fla, USA, 1991.

[3] P. Sahoo and S. K. Das, "Tribology of electroless nickel coatings—a review," *Materials & Design*, vol. 32, no. 4, pp. 1760–1775, 2011.

[4] J. N. Balaraju, T. S. N. S. Narayanan, and S. K. Seshadri, "Structure and phase transformation behaviour of electroless Ni-P composite coatings," *Materials Research Bulletin*, vol. 41, no. 4, pp. 847–860, 2006.

[5] I. Apachitei, F. D. Tichelaar, J. Duszczyk, and L. Katgerman, "Solid-state reactions in low-phosphorus autocatalytic NiP-SiC coatings," *Surface and Coatings Technology*, vol. 148, no. 2-3, pp. 284–295, 2001.

[6] W. Chen, W. Gao, and Y. He, "A novel electroless plating of Ni-P-TiO_2 nano-composite coatings," *Surface & Coatings Technology*, vol. 204, no. 15, pp. 2493–2498, 2010.

[7] J. T. Winowlin Jappes, B. Ramamoorthy, and P. Kesavan Nair, "Novel approaches on the study of wear performance of electroless Ni-P/diamond composite deposites," *Journal of Materials Processing Technology*, vol. 209, no. 2, pp. 1004–1010, 2009.

[8] Z. A. Hamid and M. T. Abou Elkhair, "Development of electroless nickel-phosphorous composite deposits for wear resistance of 6061 aluminum alloy," *Materials Letters*, vol. 57, no. 3, pp. 720–726, 2002.

[9] N. K. Shrestha, D. B. Hamal, and T. Saji, "Composite plating of Ni-P-Al_2O_3 in two steps and its anti-wear performance," *Surface & Coatings Technology*, vol. 183, no. 2-3, pp. 247–253, 2004.

[10] G.-H. Zhou, H.-Y. Ding, F. Zhou, and Y. Zhang, "Structure and mechanical properties of Ni-P-nano Al_2O_3 composite coatings synthesized by electroless plating," *International Journal of Iron and Steel Research*, vol. 15, no. 1, pp. 65–69, 2008.

[11] A. Araghi and M. H. Paydar, "Electroless deposition of Ni-P-B4C composite coating on AZ91D magnesium alloy and investigation on its wear and corrosion resistance," *Materials and Design*, vol. 31, no. 6, pp. 3095–3099, 2010.

[12] M. Mohammadi and M. Ghorbani, "Wear and corrosion properties of electroless nickel composite coatings with PTFE and/or MoS_2 particles," *Journal of Coatings Technology Research*, vol. 8, no. 4, pp. 527–533, 2011.

[13] A. Grosjean, M. Rezrazi, J. Takadoum, and P. Berçot, "Hardness, friction and wear characteristics of nickel-SiC electroless composite deposits," *Surface and Coatings Technology*, vol. 137, no. 1, pp. 92–96, 2001.

[14] H. Xu, Z. Yang, M.-K. Li, Y.-L. Shi, Y. Huang, and H.-L. Li, "Synthesis and properties of electroless Ni-P-Nanometer Diamond composite coatings," *Surface & Coatings Technology*, vol. 191, no. 2-3, pp. 161–165, 2005.

[15] D. Liu, Y. Yan, K. Lee, and J. Yu, "Effect of surfactant on the alumina dispersion and corrosion behavior of electroless Ni–P–Al_2O_3 composite coatings," *Materials and Corrosion*, vol. 60, no. 9, pp. 690–694, 2009.

[16] R. K. Roy, *A Primer on the Taguchi Method*, Society of Manufacturing Engineers, Dearborn, Mich, USA, 1990.

[17] S. K. Das and P. Sahoo, "Optimisation of tribological performance of electroless Ni-B coating using Taguchi method and grey relational analysis," *Tribology*, vol. 5, no. 1, pp. 16–24, 2011.

[18] S. Roy and P. Sahoo, "Optimization of Electroless Ni-P-W coatings for minimum friction and wear using Grey-Taguchi method," *Journal of Coatings*, vol. 2013, 13 pages, 2013.

[19] P. Gadhari and P. Sahoo, "Effect of process parameters on tribological properties of Ni-P-Al_2O_3 composite coatings," *Applied Mechanics and Materials*, vol. 592–594, pp. 1300–1304, 2014.

[20] J. Deng, "Nucleus less-data series grey modeling," *Journal of Grey System*, vol. 1, no. 1, pp. 1–24, 1989.

[21] MINITAB Incorporation, *Minitab User Manual (Release 13.2), Making Data Analysis Easier*, MINITAB Incorporation, State College, Pa, USA, 2001.