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Procedia Materials Science 5 (2014) 978 – 987

Procedia
Materials Sciencewww.elsevier.com/locate/procediaInternational Conference on Advances in Manufacturing and Materials Engineering,
AMME 2014

Comparative Study of Tribological Properties of Ni-P Coatings Under Dry and Lubricated Conditions

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Abstract

This paper presents a comparative study of tribological behavior of electroless Ni-P coating under dry and lubricated condition based on Taguchi analysis. Mild steel specimens are used as the substrate material for the deposition of Ni-P coating. Based on L_{27} orthogonal array of Taguchi analysis, the friction and wear tests are conducted on a plate-on-roller type multi-tribotester. Three process parameters viz. normal load, sliding speed and duration of sliding are considered. It is seen that the normal load is the most significant factor followed by sliding speed and sliding time at 99% confidence level in case of friction coefficient. However, for wear normal load and sliding time are significant parameters at same confidence level. Confirmation tests are carried out to validate the analysis. Finally, the surface morphology, composition and compound analysis of the coatings are done by means of scanning electron microscope, energy dispersed x-ray micro-analyzer and x-ray diffraction analyzer respectively.

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Selection and peer-review under responsibility of Organizing Committee of AMME 2014

Keywords: Electroless Ni-P coating; Friction and wear; Taguchi method; Lubrication.

1. Introduction

Electroless nickel (EN) plating is an innovative method on surface coating technology around the world. It is an autocatalytic deposition of a Ni alloy from an aqueous solution on a substrate without the application of electric current. Wurtz (1844) discovered that metallic nickel can be deposited from an aqueous solution of its salt by

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reduction with hypophosphite. Thereafter, the reaction is much studied but the metal thus formed is almost invariably in powder form. Electroless nickel (EN) coating is first developed by Brenner and Riddell (1946) to meet the challenging needs of a variety of industrial applications. These types of coating are used in industrial applications because of their excellent mechanical, electrical, physical, corrosion, hardness, friction and wear resistance properties. It provides a smooth and uniform deposition over the substrate and so it may be applied on irregular and intricate shapes of geometries also. The electroless bath typically comprises an aqueous solution of metal ions, reducing agent(s), and stabilizer(s) operating in a specific metal ion concentration, temperature, and pH ranges (Sahoo and Das, 2011).

Many researchers are attracted in the field of tribological properties. A study of wear (Staia et al., 1996) on electroless Ni-P coating reveals that the coating in as deposited state exhibits high rates of wear. Properties of coating is basically depends upon the content of phosphorous. It is observed that wear resistant increases with an increase in phosphorous content in the coating. It is also mentioned that mechanical and tribological properties of the coatings can further be improved by incorporating heat treatment temperature (Agarwala and Agarwala, 2003). It is also found on abrasive wear resistance of electroless Ni-P coating that the heat treatment decreases the abrasive wear resistance of the coatings (Staia et al., 1997). In Ni-P coating, the content of phosphorous decreases with an increase in temperature as the crystal structure changes. But the highest hardness may be achieved if the coating is heat treated at 400°C for 1 hour (Staia et al., 1996). The friction and wear behavior of electroless nickel has been evaluated mostly under dry (non-lubricated) conditions. Use of lubricant may improve the tribological behaviour of coating. But only a few researchers are focused on this region (Liu and Tandon, 1996; Panagopoulos, 2000; Chen et al., 2002; Ger et al., 2002; Wang et al., 2006).

In the present study, mild steel (AISI 1040) is taken as substrate material and deposition of Ni-P coatings is done to prepare the samples. Friction and wear characteristics are studied in a multi-tribotester varying three process parameters viz. normal load, sliding speed and sliding time based on L_{27} orthogonal array (OA). Taguchi technique is used to find out the optimum combination of process parameters for minimum friction and wear. A comparative study is carried out between dry and lubricated sliding conditions for friction and wear properties of Ni-P coatings. Confirmation tests are also done to validate the experimental results. Analysis of microstructure, composition and compounds are discussed by results of scanning electron microscope (SEM), energy dispersed x-ray micro-analyzer (EDX), and x-ray diffraction analyzer (XRD) respectively.

2. Taguchi method

Taguchi analysis is a powerful tool for design of high quality systems based on orthogonal array (OA) (Roy, 1990). It is nothing but the shortest possible matrix of combinations in which all the parameters are varied at the same time and their effect and performance interactions are studied simultaneously. Taguchi method uses a statistical measure of performance called signal-to-noise (S/N) ratio, which is logarithmic function of desired output to serve as objective functions for optimization. The S/N ratio considers both the mean and the variability into account. It is defined as the ratio of the mean (signal) to the standard deviation (noise). The ratio depends on the quality characteristics of the product/process to be optimized. The three categories of S/N ratios are used: lower-the-better (LB), higher-the-better (HB) and nominal-the-best (NB). The parameter level combination that maximizes the appropriate S/N ratio is the optimal setting. For both the cases of minimization of friction and minimization of wear, LB characteristic needs to be used. Furthermore, analysis of variance (ANOVA) (Montgomery, 2001) is performed to find the significant parameters that affect responses. Finally, a confirmation test is done to verify the optimal process parameters obtained from the parameter design.

3. Experimental details

3.1 Coating deposition

Mild steel (AISI-1040) specimens of size 20mm x 20mm x 8mm are used as the substrate material for the deposition of the electroless Ni-P coating. For preparation of samples, some machining operations are used like shaping, parting, milling and grinding. The samples are cleaned from foreign matter and corrosion products by wiping. For coating deposition the surfaces of the specimens are cleaned carefully using distilled water, etched with

50% hydrochloric acid (for 1 min), rinsed in distilled water and methanol subsequently. Finally, the cleaned samples are activated in palladium chloride at 55°C temperature for a few seconds and placed in the electroless bath. Bath composition and operating conditions are displayed in Table 1. These conditions are chosen as optimum combination of coating parameters (Sahoo and Pal, 2007). Deposition parameters are kept constant for each specimen so that the coating thickness remains constant. SEM result confirms the deposition thickness is around 32µm. After the deposition, the samples are taken out of the bath and washed in distilled water. By using a box furnace the samples are heat treated (annealed at 400°C for 1h) individually followed by normal air cooling.

3.2 Design factors and response variables

The first target is to find out the design factor along with their levels. Here the compositions and operating conditions for electroless Ni-P coating are selected through the review of literatures (Sahoo and Pal, 2007; Sahoo, 2008; Sahoo, 2009). The most important parameters are normal load (L), sliding speed (S) and duration of sliding (T). Table 2 displays the main design factors along with their levels.

Table 1. Bath composition and operating conditions.

Bath composition	Unit	Operating condition	
Nickel chloride and Nickel sulphate (1:1)	30 g/L	pH	4.5
		Time	2 h
Sodium hypophosphite	10 g/L	Dep. Temp.	80°C
Sodium succinate	12 g/L	Bath Vol.	200 ml

Table 2. Design factors along with their levels.

Design factors	Unit	Levels		
		1	2	3
Load (L)	N	50	75 ^a	100
Speed (S)	RPM	60	70 ^a	80
Time (T)	Min.	5	10 ^a	15

^a Initial testing condition

3.3 Design of Experiment

In this study, an L₂₇ 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 to fulfil the experiment. To check the DOFs in the experimental design, for three level test, the three main factors take 6 [3 × (3 – 1)] DOFs. The DOF for three second-order interactions (L × S, L × T, S × T) are 12 [3 × (3 – 1) × (3 – 1)] and so the total DOFs required is 18. As per Taguchi method, the total DOFs of selected OA must be greater than or equal to the total DOFs required for the experiment and hence L₂₇ OA has been selected. Table 3 shows in details of design factors and their interactions. 1st, 2nd and 5th column are assigned to the design factors (normal load, sliding speed and duration of sliding) simultaneously. Six columns are assigned for two-way interactions of the design factors and remaining four columns are as error terms.

3.4 Friction and wear tests

The friction and wear tests are carried out by utilizing the combination of testing parameters of a plate-on-roller type multi-tribotester (TR-25, Ducom, India) like normal load, sliding speed (rpm) and time of operation at room temperature (33°C). For tribological test under lubricated condition, commercial engine oil, Servo PRIDE-40, a product of Indian Oil is used. Here EN-coated specimen (40–45 HRc) is used as stationary plate. This plate is pressed against a rotating roller (ϕ 50mm X 20mm in size) of EN8 material (55HRc). A 1:5 ratio loading lever is used to apply normal load on the specimen with the help of loading pan. Normal load, frictional force and depth of wear are measured by sensors. The experiments are conducted with varying the design factors and levels (Table 2). Finally, the experimental results for friction coefficient and depth of wear (both for dry and lubricated condition) are recorded and shown in Table 4.

4. Results and discussion

The experimental results for friction and wear properties under dry (Sahoo and Pal, 2007) and lubricated conditions are presented in Table 4. It is to be mentioned here that the maximum wear depth is less than the average coating thickness (31.69 µm). The experimental results are analysed using Taguchi method and for this, Minitab

software (Minitab user manual, 2001) is used.

Table 3. L_{27} orthogonal array

Trial No.	Column numbers												
	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

4.1. Analysis of Signal-to-Noise ratio

In Taguchi method, the term ‘signal’ represents the desirable value (mean) for the output characteristic and term ‘noise’ represents the undesirable value (standard deviation) for the output characteristic. Therefore, S/N ratio is the ratio of the mean and the standard deviation (SD). Taguchi method uses S/N ratio to measure the quality characteristic deviating from the desired value. In this study, lower-the-better (LB) quality characteristic is used as follows:

S/N ratio for lower–the–better (minimize), S/N_S :

$$= -10 \log \left(\frac{1}{n} \sum_{i=1}^n y^2 \right) \quad (1)$$

where, y is the observed data and n is the number of observations and y is the data. These S/N ratios are expressed on a decibel scale (dB).

The response tables for these analyses are shown in Table 5 and Table 6 for friction and wear respectively. The corresponding main effects plots for coefficient of friction in lubricated condition and dry condition for same testing parameters are also shown in Fig. 1(a), 1(b), 2(a) and 2(b), respectively. In the plot for particular parameter, if the line for a 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. Optimum conditions of the control factors can be very easily determined from these response tables and main effects plots. So, the optimal tribotesting parameters combination for friction is found to be L3S3T3 (highest levels of load, speed and time) and L3S3T1 (highest levels of load and speed and the lowest level of time) for lubricated condition and dry condition respectively. For wear characteristics, optimum parameter combinations are found to be as L3S1T3 and L3S1T3 for lubricated and dry condition respectively.

From the main effects plots (Fig. 1 and Fig. 2), it is also seen that friction and wear behaviours follow almost same trend under lubricated and dry condition.

Table 4. Experimental results for Lubricated and Dry condition at their respective testing parameter combination

Exp No.	Testing Parameters			Lubricated condition		Dry condition	
	Load (N)	Speed (RPM)	Time (min)	Friction coefficient	Wear (µm)	Friction coefficient	Wear (µm)
1	50	60	5	0.084	11.841	0.257	13.57
2	50	60	10	0.082	12.870	0.264	15.65
3	50	60	15	0.078	13.952	0.276	17.22
4	50	70	5	0.067	11.931	0.228	11.21
5	50	70	10	0.067	12.401	0.247	14.19
6	50	70	15	0.065	13.850	0.256	16.24
7	50	80	5	0.055	12.213	0.172	11.16
8	50	80	10	0.052	13.296	0.179	12.97
9	50	80	15	0.052	13.342	0.211	16.06
10	75	60	5	0.053	15.138	0.114	14.15
11	75	60	10	0.051	16.672	0.149	18.31
12	75	60	15	0.048	17.855	0.163	23.58
13	75	70	5	0.047	15.891	0.095	13.54
14	75	70	10	0.047	16.968	0.099	16.56
15	75	70	15	0.046	17.164	0.109	23.16
16	75	80	5	0.043	15.668	0.077	13.38
17	75	80	10	0.042	16.526	0.090	15.79
18	75	80	15	0.043	16.621	0.094	21.75
19	100	60	5	0.041	17.502	0.072	23.07
20	100	60	10	0.036	18.334	0.073	25.78
21	100	60	15	0.037	18.817	0.076	26.39
22	100	70	5	0.039	17.343	0.055	22.33
23	100	70	10	0.035	17.557	0.059	23.46
24	100	70	15	0.032	18.218	0.064	26.30
25	100	80	5	0.028	16.545	0.042	21.91
26	100	80	10	0.024	17.531	0.044	22.19
27	100	80	15	0.024	18.311	0.049	24.46

Table 5. Response table of mean S/N ratio for coefficient of friction (a) for lubricated condition and (b) for dry condition

(a)				(b)			
Level	L	S	T	Level	L	S	T
1	23.63	25.38	26.28	1	12.79	17.08	19.68
2	26.64	26.41	26.79	2	19.41	18.92	18.96
3	29.81	28.29	27.00	3	24.72	20.93	18.28
Rank	1	2	3	Rank	1	2	3
Delta	6.18	2.91	0.72	Delta	11.93	3.85	1.4

Total mean S/N ratio of friction coefficient = 26.69 dB

Total mean S/N ratio of friction coefficient = 18.97 dB

Table 6. Response table of mean S/N ratio for wear (a) for lubricated condition and (b) for dry condition

(a)				(b)			
Level	L	S	T	Level	L	S	T
1	-22.17	-23.92	-23.36	1	-22.98	-25.66	-23.76
2	-24.34	-23.82	-23.88	2	-24.81	-25.04	-25.04
3	-25.00	-23.77	-24.26	3	-27.58	-24.68	-26.57
Rank	1	3	2	Rank	1	3	2
Delta	2.83	0.15	0.89	Delta	4.60	0.98	2.81

Total mean S/N ratio of wear = -23.84 dB

Total mean S/N ratio of wear = -25.12 dB

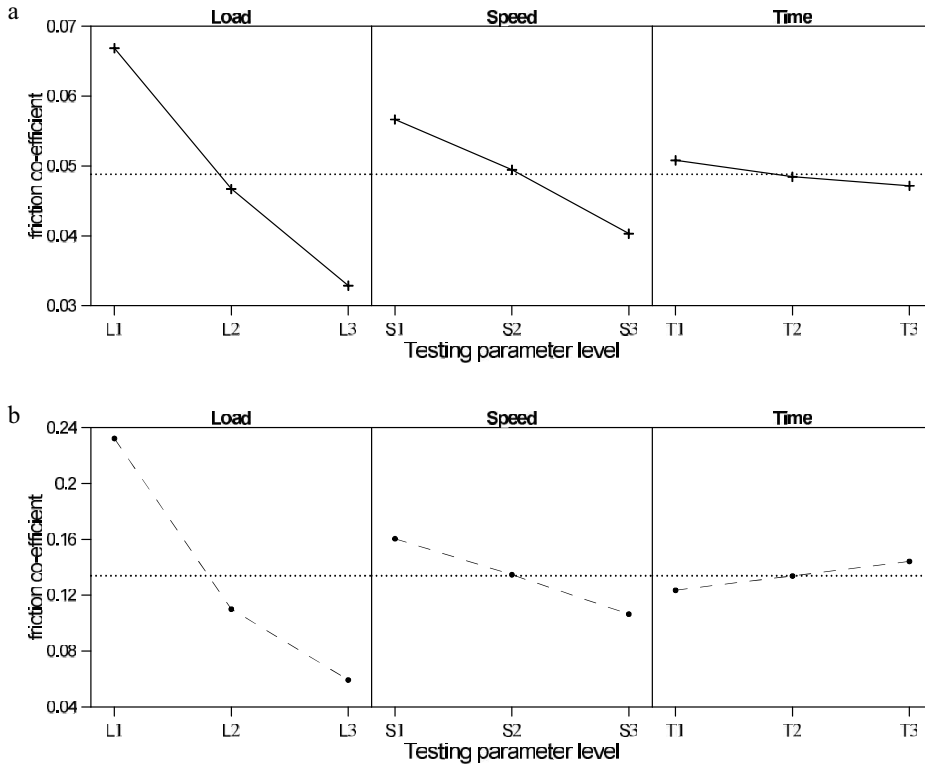
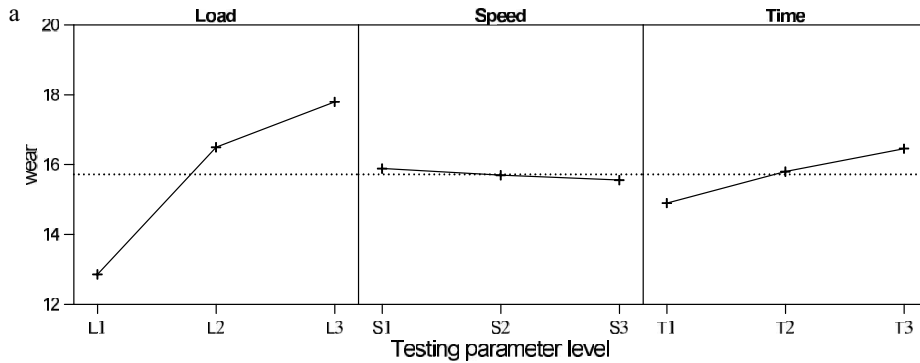


Fig.1. Main effects plot for friction coefficient (a) under lubricated condition and (b) under dry condition



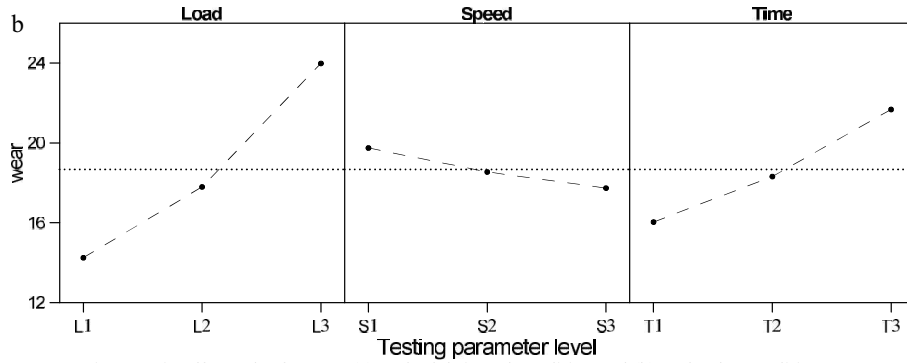


Fig.2. Main effects plot for wear (a) under lubricated condition and (b) under dry condition

4.2 Analysis of variance (ANOVA)

ANOVA, a statistical technique (Roy, 1990; Montgomery, 2001) is used to find out the significance of individual process parameters and their respective percentages of contribution. ANOVA calculates F-ratio, which is the ratio between the regression mean square and the mean square error. This ratio 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. If the calculated value of the F-ratio is higher than the tabulated value of the F-ratio, then the factor is significant at a desired significance level. In general, when the F value increases the significance of the parameter also increases.

Table 7. ANOVA table for coefficient of friction (a) for lubricated condition and (b) for dry condition

(a)						(b)					
Source	DF	SS	MS	F _{calculated}	% P	Source	DF	SS	MS	F _{calculated}	% P
L	2	172.088	86.044	1571.58*	77.64	L	2	642.9380	321.4690	1367.23*	88.60
S	2	39.231	19.616	358.27*	17.70	S	2	66.7630	33.3815	141.97*	9.20
T	2	2.506	1.253	22.89*	1.13	T	2	8.7630	4.3815	18.63*	1.21
L*S	4	6.059	1.515	27.67*	2.73	L*S	4	3.9110	0.9778	4.16	0.54
L*T	4	0.226	0.057	1.03	0.10	L*T	4	1.1700	0.2925	1.24	0.16
S*T	4	1.106	0.277	5.05	0.50	S*T	4	0.2450	0.0613	0.26	0.03
Error	8	0.438	0.055		0.20	Error	8	1.8810	0.2351		0.26
Total	26	221.654			100.00	Total	26	725.6710			100.00

Tabulated values for variance ratio (F) at 99% confidence level (F_{0.01, 2, 8} = 8.6491 F_{0.01, 4, 8} = 7.0060)

* - Significant parameter

Table 8. Response table of ANOVA for wear (a) for lubricated condition and (b) for dry condition

(a)						(b)					
Source	DF	SS	MS	F _{calculated}	% P	Source	DF	SS	MS	F _{calculated}	% P
L	2	39.5755	19.7878	460.85*	89.24	L	2	96.4209	48.2105	807.38*	65.91
S	2	0.1029	0.0515	1.20	0.23	S	2	4.4199	2.2100	37.01*	3.02
T	2	3.626	1.8130	42.22*	8.18	T	2	35.6953	17.8477	298.89*	24.40
L*S	4	0.2192	0.0548	1.28	0.49	L*S	4	0.4033	0.1008	1.69	0.28
L*T	4	0.2398	0.0600	1.40	0.54	L*T	4	8.2473	2.0618	34.53*	5.64
S*T	4	0.2408	0.0602	1.40	0.54	S*T	4	0.6346	0.1587	2.66	0.43
Error	8	0.3435	0.0429		0.77	Error	8	0.4777	0.0597		0.33
Total	26	44.3477			100.00	Total	26	146.2990			100.00

Tabulated values for variance ratio (F) at 99% confidence level (F_{0.01, 2, 8} = 8.6491 F_{0.01, 4, 8} = 7.0060)

* - Significant parameter

ANOVA results for friction coefficients are presented in Table 7. It is seen that the most significant parameter is applied load followed by sliding speed and sliding duration for both dry and lubricated conditions. Under lubricated condition, the interaction between applied load and sliding speed is also significant.

From ANOVA table (Table 8) for wear under lubricated condition, only applied load and sliding time are found as significant parameter. But for dry condition, it is seen that the most significant parameter is applied load followed by sliding time, sliding speed and interaction between applied load and sliding time.

4.3 Confirmation test

After the optimal level of testing parameters has been found out, it is necessary that verification tests are to be carried out in order to control the accuracy of analysis and to validate the experimental results. The estimated S/N ratio ($\hat{\eta}$) can be calculated as:

$$\hat{\eta} = \eta_m + \sum_{i=1}^o (\bar{\eta}_i - \eta_m) \quad (2)$$

where, η_m is the total mean S/N ratio, $\bar{\eta}_i$ is the mean S/N ratio at the optimal testing parameter level and o is the number of the main design testing parameters that significantly affect the friction and wear characteristics of electroless Ni-P coating.

Table 9 shows the results for the estimated friction coefficient and the actual friction coefficient using the optimal parameters under lubricated condition. The improvement of S/N ratio from initial to optimal condition is 5.8996 dB which means co-efficient of friction is reduced by 22.27%. Similarly, Table 10 shows the results for the estimated and actual wear using the optimal parameter setting under lubricated condition. The improvement of S/N ratio from initial to optimal condition is 2.8565 dB i.e. wear is reduced by 11.62%. It is seen that there is a good agreement between the estimated and actual value for both the cases.

Table 9. Result of confirmation test for friction coefficient.

	Initial parameter	Optimal parameter	Exp.
Level	L2S2T2	L3S3T3	
Friction coefficient	0.047		0.024
S/N ratio (dB)	26.4962	31.7156	32.3958

Improvement of S/N ratio = 5.8996 dB.

Table 10. Results of confirmation test for wear

	Initial parameter	Optimal parameter	Exp.
Level	L2S2T2	L1S3T1	
Wear (μm)	16.968		12.213
S/N ratio (dB)	-24.5926	-21.6200	-21.7361

Improvement of S/N ratio = 2.8565 dB

4.4 Surface morphology and composition study

These studies have been done to analysis the microstructure (from SEM, FEI Quanta 200), composition (from EDX, FEI Quanta 200) and compound (from XRD, Rigaku, Miniflex) of the deposited coating before and after annealing at 400°C in order to observe the effect of heat treatment.

From the study of SEM micrographs of these surfaces it is seen that there are many globular particles on the surface of the substrate. Larger (for Ni) and smaller (for P) spherical nodular structures represent on surface. The surface is optically smooth and of low porosity and no surface damage is found. This analysis indicates that the as-deposited Ni-P film is a mixture of amorphous and microcrystalline phases. When Ni-P deposits are heat treated both their atomic structure and microstructure undergo modification. Both the microcrystalline and the amorphous deposits undergo a crystal growth process and such heat treatment results in a mixture of relatively coarse-grained structure. SEM micrographs of electroless Ni-P deposit for as deposited and after heat treatment are shown in fig. 3(a) and 3(b) respectively. The surface is optically smooth having low porosity and no surface damage.

EDX analysis shows that the crystal structure of coating changes from amorphous (90.75% Ni and 09.25% P) to crystalline (93.18% Ni and 06.82% P) structure due to annealing. EDX patterns of as plated sample and heat treated sample are also shown in Fig. 4(a) and Fig. 4(b).

XRD analysis also confirms that coating is amorphous in as-plated condition and so peaks are not easily identified.

But after heat treatment, deposits crystallize and easily found three major compound peaks (Ni_3P_2 , Ni_2P and NiP_2). Fig. 5(a) and Fig. 5(b) represent XRD plots for as deposited and heat treated condition respectively.

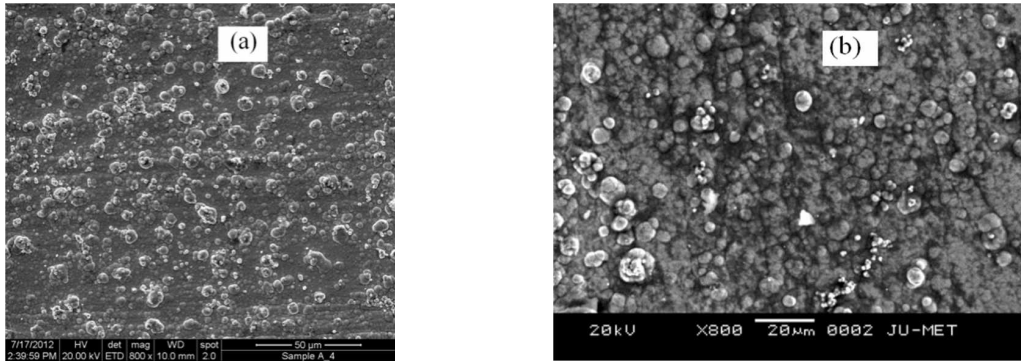


Fig.3. SEM micrographs of electroless Ni-P deposit in (a) as-deposited and (b) heat treated sample.

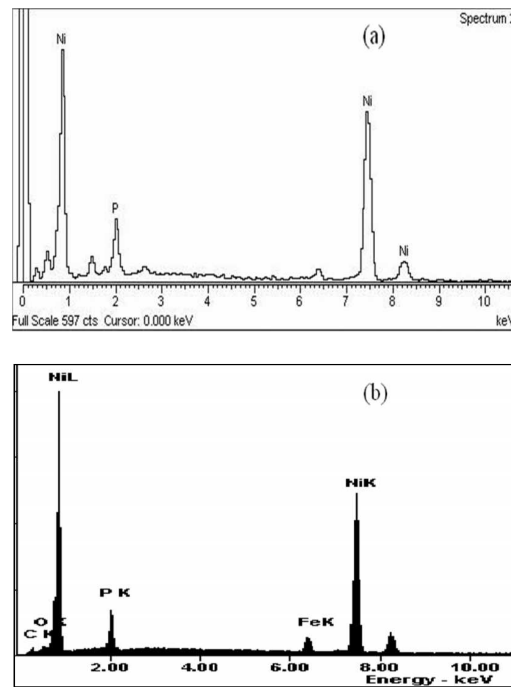


Fig.4. EDX pattern of (a) as-deposited and (b) heat treated sample

6. Conclusion

Taguchi method is used to optimize the tribological testing parameters of EN coatings for friction and wear properties. It is seen from the study that the normal load is the most significant factor followed by sliding speed and sliding time at 99% confidence level in case of friction coefficient. However, for wear normal load and sliding time are significant parameters at same confidence level. The optimum combination of process parameters are found out for minimum friction and wear from the study. It is seen that minimum friction is found out at the highest levels of normal load, speed and sliding time under lubricated condition. Under dry condition, minimum friction is found at the highest levels of normal load and speed and the lowest level of sliding time. Minimum wear is found at the

highest levels of normal load and time and at the lowest level of sliding speed for both dry and lubricated condition. Confirmations tests are carried out to validate the analysis. Finally, the surface morphology, composition and compound analysis of the coatings are done by means of scanning electron microscope, energy dispersed x-ray micro-analyzer and x-ray diffraction analyzer respectively.

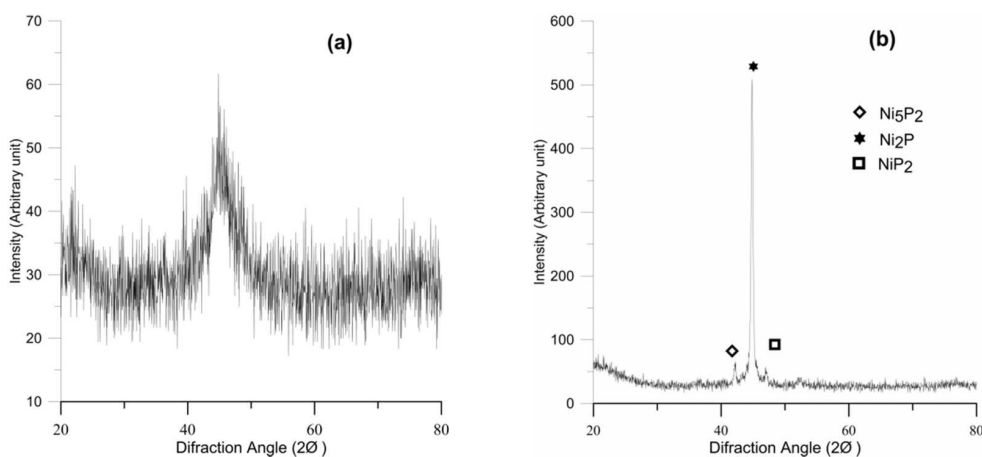


Fig.5. XRD patterns of electroless Ni-P deposit in (a) as-deposited and (b) heat treated sample.

Acknowledgements

The authors gratefully acknowledge the partial financial support of University Grants Commission (UGC), Govt. of India through Major Research Project vide Ref. No. F No. 41-984/2012 (SR) dated 25.07.2012.

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