IOSR Journal of Mechanical and Civil Engineering (IOSR-JMCE)
e-ISSN: 2278-1684, p-ISSN: 2320-334X
PP 22-26
www.iosrjournals.org

Study of Tribological Properties of Ni-P Coatings Under Lubrication

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ABSTRACT : *This work presents an experimental study of tribological performance of electroless Ni-P coating under lubricated condition using grey relational analysis. Mild steel specimens are used as the substrate material for the deposition of Ni-P coating. Tribological tests are carried out based on L_{27} orthogonal array(OA) of Taguchi analysis varying three test parameters viz. normal load, sliding speed and sliding time in a multi-tribotester using block-on-roller configuration in lubricated condition. The experimental results for friction coefficient and wear are analysed using grey relational analysis coupled with Taguchi analysis. An optimal test parameter combination is found out for minimum wear and friction coefficient. It is seen that the normal load is the most significant factor followed by sliding speed and sliding time at 99% confidence level. Finally, a confirmation test is carried out to validate the analysis. The surface morphology and composition analysis of the coatings are done by means of scanning electron microscope (SEM) and energy dispersed x-ray micro-analyzer (EDX) respectively.*
Keywords: *Friction and wear, Grey relational analysis, Lubrication, Ni-P coating, Optimization.*

I. INTRODUCTION

Electroless nickel (EN) plating is an innovative method on surface coating technology. It is an autocatalytic deposition of a Ni-P alloy from an aqueous solution on a substrate without the application of electricity. Electroless Ni-P coating [1] is first developed by Brenner and Riddell [2] in 1946 to meet the challenging needs of a variety of industrial applications. Coatings are used in industrial applications due to their excellent mechanical, electrical, physical, corrosion, hardness, friction and wear resistance properties. Intricate parts or geometries can also be used as substrate to deposit EN coating due to its smooth and uniform deposition property. 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 [3].

Many researchers have focussed on the analysis of tribological properties of EN coatings. A study of wear on electroless Ni-P coating in dry condition reveals that the coating in the as-deposited state exhibits high rates of wear. Properties of coating basically depend upon the content of phosphorous and wear resistance increases with increase of phosphorous content in the coating [4]. It is found that heat treatment changes the abrasive wear resistance of the coatings [5]. In Ni-P coating, the content of phosphorous decreases with an increase in temperature but highest hardness may be achieved if the coating is heat treated at 400°C for 1 hour [6]. The friction and wear behaviour of electroless nickel has been evaluated mostly under dry (non-lubricated) conditions and two mechanisms viz. adhesion and abrasion are quoted as mechanisms involved in the failure of these coatings [7].

The use of lubricant improves the tribological behaviour of coating and few researchers have focused on this aspect [8-16]. In the present study, friction and wear characteristics are studied in a multi-tribotester under lubricated condition varying three test parameters viz. normal load, sliding speed and sliding time. Samples are prepared by depositing Ni-P coatings on mild steel (AISI 1040) substrates and then heat treatment (annealed) is done at 400°C for 1 hour. Based on Taguchi's L_{27} orthogonal array (OA), experiments are carried out. Grey relational grade coupled with Taguchi analysis is used to find out the optimum combination of test parameters for multiple responses viz. coefficient of friction and wear. Confirmation test is also done to validate the analysis. Finally, analysis of microstructure and composition are done using SEM and EDX respectively.

II. EXPERIMENTAL DETAILS

Mild steel (AISI-1040) specimens of size 20mm x 20mm x 8mm are used as the substrate material for the deposition of EN coating. The samples are cleaned from foreign matter and corrosion products by wiping. Then, for coating deposition the surfaces of the specimens are cleaned 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 and placed in the bath. Bath composition and operating conditions [14] are shown in Table 1. Deposition parameters are kept constant to maintain same coating thickness (≈ 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) separately followed by air cooling.

Table 1. Bath composition and operating conditions.

Bath composition		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

For tribological tests, three test parameters viz. normal load (L), sliding speed (S) and sliding time (T) are considered as design factors and coefficient of friction and wear are considered as the responses. Table 2 displays the main design factors along with their levels. In this study, an L_{27} orthogonal array (OA), which has 27 rows corresponding to the number of tests (26 degrees of freedom) with 13 columns at three levels, is chosen to fulfil the experiment. Commercially available lubricant, Servo PRIDE-40 (a product of Indian Oil) is used in this study. The friction and wear tests are carried out by utilizing the combination of test parameters in a block-on-roller type multi-tribotester (Ducom, India) at room temperature (33°C) under lubrication. Here EN-coated specimen (20mm X 20mm X 8mm) with a hardness value of approximately 30-40 HRc is placed as stationary plate and the rotating roller (ϕ 50mm X 20mm, material-EN8) with 55HRc is placed at the bottom. The experimental results for friction coefficient and wear depth are recorded and shown in Table 3.

Surface morphology and composition studies have been conducted using FEI Quanta 200.

III. RESULTS AND DISCUSSION

Grey relational analysis coupled with Taguchi analysis is used here for multiple response optimization. Grey theory was established by Deng [17], which includes Grey relational analysis of a system. This analysis is very much useful for multi response analysis. Taguchi analysis [18] can be used for single response optimization. But the optimization of multiple performance characteristics is different from that of a single performance characteristic. The lower signal to noise ratio (S/N ratio) for one performance characteristic may correspond to a higher S/N ratio for another. Therefore, the overall evaluation of the S/N ratio is required for the optimization of multiple performance characteristics. To eliminate such problem, the grey relational analysis is necessary and hence utilized in this study. Experimental results and grey relational analysis for friction coefficient and wear are shown in Table 3.

The analysis of signal to noise (S/N) ratio is done using the LB (lower-the better) criterion with the grey relational grade as the performance index. The response table for this analysis is shown in Table 4. The corresponding main effects plot is also shown in Fig. 1. In the plot for particular parameter, if a line has the highest inclination then that parameter is the most significant parameter. It is seen that parameters L (normal load), S (sliding speed) and sliding time (T) are significant parameters. The optimal test parameter combination for minimum friction and wear is found to be L1S3T1.

Table 3. Experimental results and grey relational analysis for friction coefficient and wear.

Exp. No.	Experimental results		Gray relational analysis							
			Normalized data		Values of Δ_{oi}		relational coefficient		Grade	Order
	Friction coefficient	Wear (μm)	Friction coefficient	Wear	Friction coefficient	Wear	Friction coefficient	Wear		
1	0.084	11.84	0.000	1.000	1.000	0.000	0.333	1.000	0.667	5
2	0.082	12.87	0.033	0.853	0.967	0.147	0.341	0.772	0.557	11
3	0.078	13.95	0.100	0.697	0.900	0.303	0.357	0.623	0.490	23
4	0.067	11.93	0.283	0.987	0.717	0.013	0.411	0.975	0.693	2
5	0.067	12.40	0.283	0.920	0.717	0.080	0.411	0.862	0.636	7
6	0.065	13.85	0.317	0.712	0.683	0.288	0.423	0.635	0.529	15

7	0.055	12.21	0.483	0.947	0.517	0.053	0.492	0.904	0.698	1
8	0.052	13.30	0.533	0.791	0.467	0.209	0.517	0.706	0.611	8
9	0.052	13.34	0.533	0.785	0.467	0.215	0.517	0.699	0.608	9
10	0.053	15.14	0.517	0.527	0.483	0.473	0.508	0.514	0.511	21
11	0.051	16.67	0.550	0.307	0.450	0.693	0.526	0.419	0.473	26
12	0.048	17.86	0.600	0.138	0.400	0.862	0.556	0.367	0.461	27
13	0.047	15.89	0.611	0.419	0.389	0.581	0.562	0.463	0.513	20
14	0.047	16.97	0.611	0.265	0.389	0.735	0.562	0.405	0.484	25
15	0.046	17.16	0.639	0.237	0.361	0.763	0.581	0.396	0.488	24
16	0.043	15.67	0.683	0.451	0.317	0.549	0.612	0.477	0.545	13
17	0.042	16.53	0.700	0.328	0.300	0.672	0.625	0.427	0.526	17
18	0.043	16.62	0.683	0.315	0.317	0.685	0.612	0.422	0.517	18
19	0.041	17.50	0.717	0.189	0.283	0.811	0.638	0.381	0.510	22
20	0.036	18.33	0.800	0.069	0.200	0.931	0.714	0.349	0.532	14
21	0.037	18.82	0.783	0.000	0.217	1.000	0.698	0.333	0.516	19
22	0.039	17.34	0.750	0.211	0.250	0.789	0.667	0.388	0.527	16
23	0.035	17.56	0.817	0.181	0.183	0.819	0.732	0.379	0.555	12
24	0.032	18.22	0.867	0.086	0.133	0.914	0.789	0.354	0.572	10
25	0.028	16.55	0.933	0.326	0.067	0.674	0.882	0.426	0.654	6
26	0.024	17.53	1.000	0.184	0.000	0.816	1.000	0.380	0.690	3
27	0.024	18.31	1.000	0.073	0.000	0.927	1.000	0.350	0.675	4

Table 4. Response table for means of grey relational grade

Level	L	S	T
1	0.6099	0.5240	0.5908
2	0.5019	0.5552	0.5627
3	0.5812	0.6138	0.5395
Rank	1	2	3
Delta	0.1079	0.0898	0.0512

Total mean grey relational grade = 0.5643 dB

ANOVA [18, 19] is a statistical technique to find out the significance of individual test parameters and their interactions on the system response under consideration and also their respective percentage of contributions. This is done by separating the total variability of the S/N ratio. Minitab [20] is used for the analysis and result is shown in Table 5. It is seen that at 99% confidence level the most significant parameters are normal load (L) followed by sliding speed (S), sliding time (T) and the interactions of L with T and L with S.

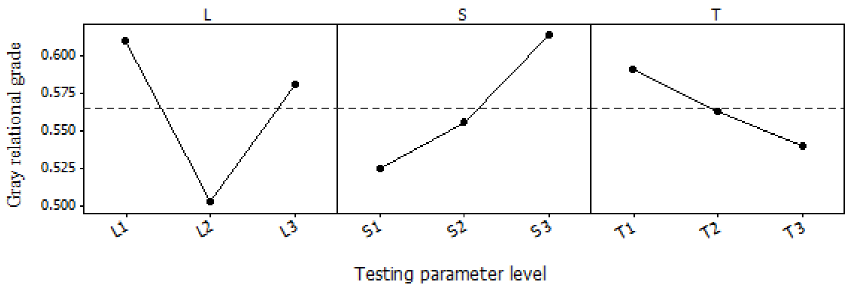


Fig. 1 Main effects plot for grey relational grade

Table 5. Results of ANOVA for grey relational grade

Source	DOF	SS	MS	$F_{calculated}$	$F_{tabulated}$	P
L	2	0.056	0.028	73.18*	8.6491	38.54
S	2	0.037	0.019	48.69*	8.6491	25.64
T	2	0.012	0.006	15.42*	8.6491	8.12
LxS	4	0.013	0.003	8.51*	7.0060	8.97
SxT	4	0.001	0.000	0.97	7.0060	1.02
LxT	4	0.023	0.006	14.81*	7.0060	15.60

Table 6. Results of confirmation test

	Initial parameter	Optimal parameter	Exp.
Level	L2S2T2	L1S3T1	
Wear	16.968		12.213
Friction coefficient	0.047		0.055
Grade	0.4837	0.6859	0.6978

Error	8	0.003	0.000			2.11

*- significant parameter.

Improvement of grey relational grade = 0.2141.

In order to evaluate the accuracy of the analysis and to validate the results, a confirmation test is carried out and presented in Table 6. It is seen that the improvement of grey relational grade from initial to optimal level is 0.2141 (44.26%). So, it is a highly significant improvement.

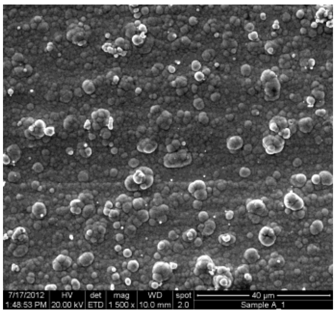
SEM micrograph of the coating surface confirms that there are many globular particles (larger for Ni and smaller for P). The surface is optically smooth and no surface damage is found as shown in Fig 2(a). The surface of the Ni-P coating appears to be dense. Fig. 2(b) shows the EDX analysis of the heat-treated sample and it is seen that the peaks of nickel and phosphorous are quite specific.

IV. CONCLUSION

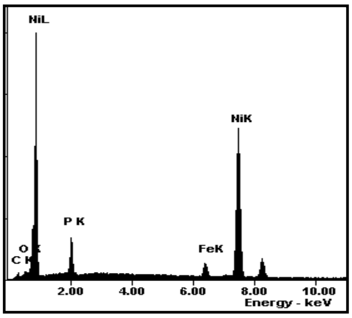
Taguchi OA with grey relational analysis is used to optimize the tribological testing parameters of electroless Ni-P coating. It is seen that the most significant parameter at 99% confidence level is normal load followed by sliding speed, sliding time and the interactions of load with time and load with speed. In the specified test range, the combination of lower load, higher speed and lower sliding time shows minimum friction and wear. A confirmation test is carried out to validate the analysis and there is a considerable amount of improvement from the initial setting to the optimal setting. Finally, the surface morphology and composition analysis of the coatings are done using scanning electron microscope (SEM) and energy dispersed x-ray micro-analyzer (EDX) respectively.

V. 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.



(a)



(b)

Fig. 2 (a) SEM micrograph of the as-deposited sample; (b) EDX pattern of the heat-treated sample

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