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Friction performance of electroless Ni-P coatings in alkaline medium and optimization of coating parameters

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Abstract

The present paper studies the friction performance of electroless Ni-P coating in alkaline medium (10 % NaOH solution) and optimization of the coating process parameters is performed for minimum friction using Taguchi method based on L_{27} orthogonal array. The study is carried out using different combinations of four coating process parameters, namely, concentration of nickel source (A), concentration reducing agent (B), bath temperature (C) and annealing temperature (D). The friction tests are conducted with a pin-on-disk tribometer. The optimum combination of process parameters for minimum friction is obtained. Also, analysis of variance (ANOVA) is performed to find out the significant contribution of each coating process parameters and their interactions. ANOVA reveals that bath temperature has the maximum contribution in controlling the friction behaviour of Ni–P coating. The surface morphology and composition of coatings are studied with the help of scanning electron microscopy (SEM), energy dispersed X-ray (EDX) analysis and X-ray diffraction (XRD) analysis. It is found that the Ni-P coating is amorphous in as-deposited condition but gradually turns crystalline with heat treatment.

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Keywords: Electroless Ni-P coating, friction, alkaline medium, Taguchi method, optimization.

1. Introduction

Electroless plating deposition was proposed by Brenner and Riddell in the middle of the last century [1,2]. Electroless nickel coating is a process for depositing a nickel alloy from aqueous solutions onto a substrate without

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the use of electric current. Electroless nickel coating is a chemical process which reduces nickel ions in solution to nickel metal by chemical reduction. Some of the unique properties of electroless nickel coating, such as thickness uniformity, hardness, corrosion resistance, tribological and magnetic response have resulted in its use in many different industries [3-5]. The properties and microstructures of electroless Ni-P (EN) coatings depend on the amount of phosphorous alloyed in the deposit [6,7]. The mechanical and tribological properties of these coatings can further be improved by the incorporation of hard particles [8], heat treatment [7,9] and dry lubricants [10,11]. Friction of EN coatings has been focused by many researchers. The friction study of EN coating with a ramp apparatus concluded that coatings with high phosphorus content have higher friction coefficient than medium or low phosphorus electroless coatings [12]. Electroless Ni-P coatings are used in different environmental conditions. Typically it finds wide application in chemical and petrochemical industries [13]. The present study considers the friction behavior of the coating in alkaline solution and optimization of coating process parameter for minimum friction based on Taguchi methodology. The characterization of the coating is done with the help of scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX) and X-ray diffraction (XRD) analyzer.

2. Taguchi method

Taguchi method [14,15] is employed to optimize the EN coating process parameters for minimum friction based on L_{27} orthogonal arrays (OA). Thus the time and cost of experimentation is decreased. Taguchi recommends the use of the loss function to measure the quality characteristic. The value of loss function is further converted into statistical measure which is called S/N (signal-to-noise) ratio. Taguchi analysis uses S/N ratio to convert the output response into a value for the evaluation purpose. The S/N ratio characteristic can be divided into three categories: lower the better (LB), higher the better (HB) and Nominal the best (NB). For the present study of friction, where friction is to be minimized the LB criteria is used. Furthermore, a statistical analysis of variance (ANOVA) is performed to find which process parameters are statistically significant [16]. With the S/N ratio and ANOVA analysis, the optimal combination of coating process parameters can be predicted.

3. Details of experiment

3.1. Deposition of coating

Mild steel (AISI 1040) is used as the substrate material (cylindrical pin of size 4 mm diameter ×30 mm long) for the deposition of EN films. Turning, parting and grinding process are used for the preparation of the solid cylindrical specimens. The specimens after thorough cleaning are given a pickling treatment with dilute (18%) hydrochloric acid for one minute to remove any surface layer formed like rust or other oxide. Subsequently, the samples are rinsed in distilled water followed by methanol cleaning prior to coating. The selected bath composition and operating condition of EN coatings is enlisted in Table 1.

` Table 1. Ingredients of electroless bath and their ranges.

Sl. No.	Parameters	Range of parameters
1	Nickel sulphate	15 – 25 g/l
2	Nickel chloride	15 – 25 g/l
3	Sodium hypophosphite	10 – 24 g/l
4	Sodium succinate	12 g/l
5	Deposition temperature	80 – 90^{o}C
6	pH of solution	4.5

EN deposition is performed using nickel sulphate and nickel chloride as the source of nickel, sodium hypophosphite as the reducing agent and sodium succinate as the stabilizer. The concentration of stabilizer used in baths is kept fixed. The pH value of the bath is maintained at a fixed value by adding required quantity of dilute hydrochloric acid. The cleaned samples are activated in palladium chloride at 55^{o}C temperature and placed in the bath for deposition for 2 h. For each sample the procedure is repeated twice. This is done to get considerable coating thickness which is necessary for carrying out the tribological tests on the coating. Deposition time is kept constant for all specimens so that the coating thickness remains approximately constant and the average coating thickness is found to be around 50 µm. After deposition, the samples are heat treated (at 300°C, 400°C, and 500°C) in box furnace for 1 h according to the OA.

3.2. Design of experiment

Based on the Taguchi method, an OA is employed to reduce the number of experiments for determining the optimal process parameters. An OA provides the shortest possible matrix of combinations in which all the parameters are varied to consider their direct effect as well as interactions simultaneously. There are number of design factors that can affect friction performance of EN coatings. In this investigation, design factors and their levels are shown in Table 2. It is a four-factor three-level experiment, so the total degree of freedom (DOF) considering the individual factors and their interaction is 20. Here, L_{27} OA is selected as it satisfies all the DOF conditions. The selected array requires the execution of 27 experiments. The factors (A, B, C and D) and their interactions (A×B, A×C and B×C) are assigned to their respective positions in the OA. Here, coefficient of friction (COF) of EN coating is taken as the response variable.

Table 2. Design factors and their levels

Design factors	Unit	Levels		
		1	2	3
Concentration of source of nickel (A)	(g/l	30	40	50
Concentration of reducer agent (B)	(g/l	10	17	24
Deposition temperature (C)	°C	80	85	90
Annealing temperature (D)	°C	30	40	50

3.3. Friction test

Friction performance of the EN coated specimens are carried out under alkaline medium (10 % NaOH solution) at ambient temperature of about 28°C. The friction tests are conducted in a pin-on-disk tribometer (TR-208-M2, Ducom, India). A pictorial view of the tribometer is shown in Fig. 1.

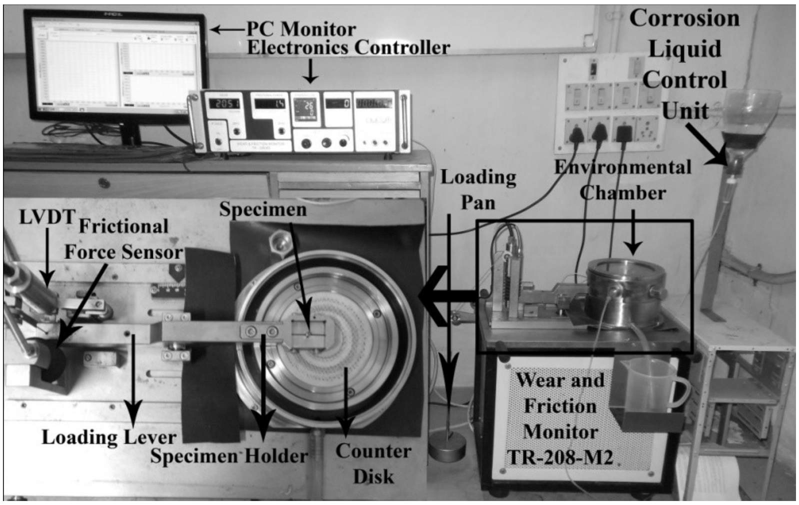

Fig. 1 Pictorial view of tribometer

The coated specimens serve as test specimens which are held perpendicular against a rotating alumina disk (100 mm dia × 8 mm thickness). Microhardness of alumina disk is 1680 HV_1, which is higher than the microhardness of EN coated specimen (average microhardness value is 700 HV_1). The average surface roughness of EN coating is 0.62-0.83 µm. Normal load is applied by placing dead weights on loading pan. The frictional force is measured by a frictional force sensor (IPA) that uses a beam type load cell of range 0 to 100 N with accuracy of 0.1 ± 2% N. The experiments are conducted with a constant load of 20 N with a wear track dia of 40 mm and at 50 rpm and for a constant time of 10 min considering the small coating thickness. Experimental data of friction test are shown in Table 3.

Table 3. Experimental results for friction coefficient and their corresponding S/N ratio

Exp. No.	COF	S/N ratio (dB)	Exp. No.	COF	S/N ratio (dB)	Exp. No.	COF	S/N ratio (dB)
1	0.315	10.0338	10	0.232	12.6902	19	0.478	6.4114
2	0.351	9.0939	11	0.347	9.1934	20	0.303	10.3711
3	0.259	11.7340	12	0.171	15.3401	21	0.332	9.5772
4	0.171	15.3401	13	0.165	15.6503	22	0.235	12.5786
5	0.549	5.2086	14	0.341	9.3449	23	0.347	9.1934
6	0.463	6.6884	15	0.500	6.0206	24	0.313	10.0891
7	0.394	8.0901	16	0.303	10.3711	25	0.109	19.2515
8	0.241	12.3597	17	0.369	8.6595	26	0.319	9.9242
9	0.473	6.5028	18	0.236	12.5418	27	0.155	16.1934

3.4. Surface morphology and composition study

Study of Surface morphology of the EN coating is obtained by SEM (JEOL, JSM-6360) in order to analyze the microstructure of the deposited coatings before and after annealing at various temperatures to see the effect of annealing temperature. SEM is also done after tribological testing to see the wear track patterns. EDX analysis (Inca, Oxford) is done in conjunction with SEM to study the composition of the Ni-P coatings in terms of the percentages of nickel and phosphorous. The different precipitated phases of the deposits both before and after annealing were analyzed by X-ray diffraction (XRD) analyzer (Rigaku, Ultima III).

4. Results and discussion

4.1. Analysis of signal-to-noise ratio

To evaluate robustness, Taguchi method needs to capture the variability within a trial condition. Hence Taguchi technique utilizes the S/N ratio approach to measure the quality characteristic deviating from the desired value. Taguchi method uses the S/N ratio approach instead of the average value to convert the experimental results into a value for the evaluation characteristic in the optimum parameter analysis. In the present work, S/N ratio analysis is done with COF as the performance index. As friction force is to be minimized, the S/N ratio is calculated using LB (Lower the better) criterion. Table 3 shows the experimental values of COF and the corresponding S/N ratio. The average of S/N ratio of each level of the factors of A, B, C and D is given in Table 4 and total mean S/N ratio of all the 27 experiment is also listed in this Table. From Table 4 and Fig. 2, it is found that process parameter C is the highest delta value (rank 1). Hence, bath temperature has done the more effective roll play for frictional force of EN coatings surface. Parameter A is also found to have some influence over the frictional force. But parameters B and D have the least influence over the friction performance of the coating. From the Fig. 3, it can be seen that there is strong interaction of parameters A Vs B and parameters B Vs C. But parameter A Vs C is not found any indicative interaction. The optimal combination of parameter is found to be A3B3C1D1.

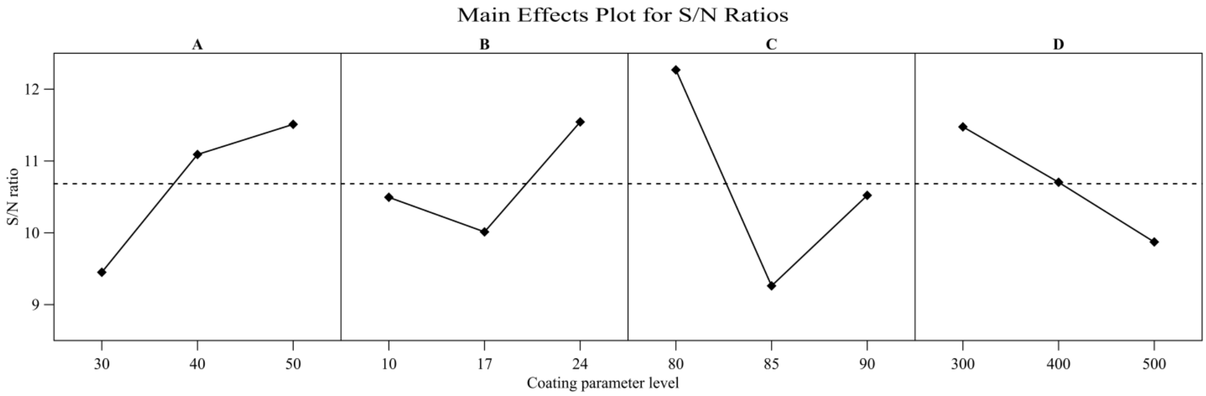

Fig. 2. Main effects plot for signal-to-noise ratio

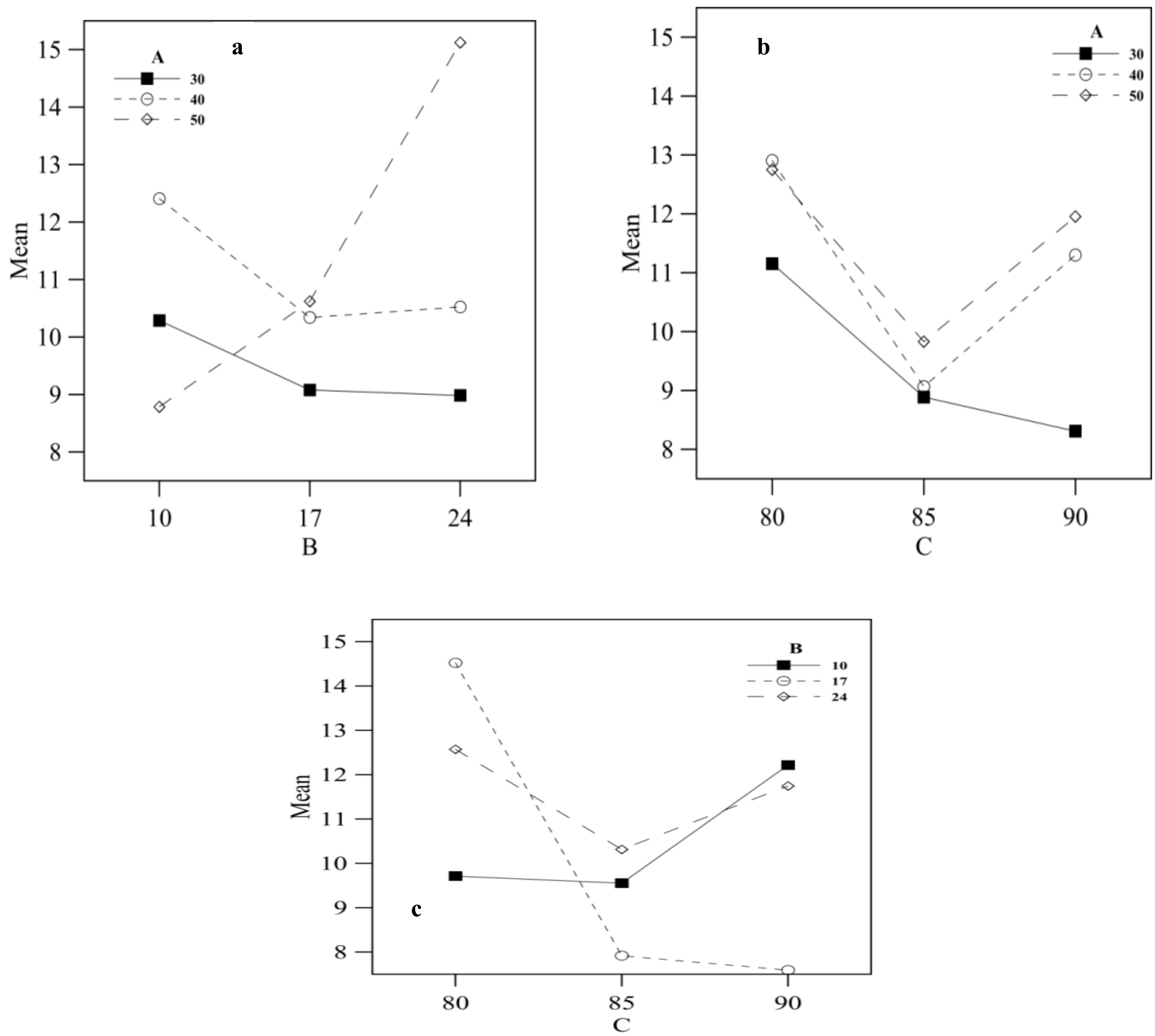

Fig. 3. Interaction plot for mean S/N ratio (a) A vs B (b) A vs C and (c) B vs C

Table 4. Response table for mean S/N ratio

Level	A	B	C	D
1	9.45	10.494	12.269	11.476
2	11.09	10.013	9.261	10.703
3	11.51	11.544	10.521	9.871
Rank	2	4	1	3
Delta	2.06	1.531	3.008	1.604

Total mean S/N ratio = 10.6835 dB

4.2. Analysis of variance (ANOVA)

The Taguchi experimental method could not judge the effect of individual parameters on the entire process, thus the idea of the analysis of variance is to find out the significance of process parameters and also the percentage contributions of the factors and the interactions in affecting the response. From Table 5, it is seen that parameter C i.e. bath temperature has got the maximum contribution in controlling friction performance of EN coating. Moreover, it is found that parameter A i.e. concentration of source of nickel also has moderate contribution in controlling the friction performance. Among the interactions, the interaction between A×B and B×C have the more contribution in controlling the friction performance of EN coating while parameter B, D and interaction A×C have almost no considerable significant influence within the experimental range considered in the study.

Table 5 Results of ANOVA for coefficient of friction

Source	DOF	SS	MS	F	Contribution (%)
A	2	21.33	10.66	0.77	6.82
B	2	11.03	5.52	0.4	3.53
C	2	41.06	20.53	1.48	13.14
D	2	11.59	5.79	0.42	3.71
A*B	4	63.78	15.95	1.15	20.41
A*C	4	8.46	2.12	0.15	2.71
B*C	4	71.85	17.96	1.29	22.99
Error	6	83.44	13.91		
Total	26	312.55			

4.3. Validation test

After the optimal level of process parameters have been found, it is necessary that validation test is carried out in order to evaluate the accuracy of the analysis and to validate the experimental results. The estimated S/N ratio, $\hat{\eta}$ using the optimal level of the process parameters can be calculated as:

$$\hat{\eta} = \eta_m + \sum_{i=1}^{o}(\overline{\eta}_i - \eta_m) \tag{1}$$

Where, η_m is the total mean S/N ratio, η_i is the mean S/N ratio at the optimal process parameter level and o is the number of main design process parameters that significantly affect the friction performance of EN coating under alkaline solution. Table 6 shows the comparison of the estimated S/N ratio with the actual S/N ratio using the optimal parameters. The increase of S/N ratio from initial coating parameter to the optimal coating parameter is 9.5187 dB, which means coefficient of friction is reduced about 67 % and it is considered as a significant improvement.

Table 6 Results of validation test

	Initial condition	Optimal condition	
		Prediction	Experimentation
Level	A2B2C2D2		A3B3C1D1
Coefficient of friction	0.365		0.122
S/N ratio (dB)	8.7541	13.0955	18.2728

Improvement of S/N ratio = 9.5187 dB

4.4. Surface morphology and compositional study

The coated as well as heat treated (at 400^{o}C) specimens are studied by SEM. Fig. 4 (a) shows the SEM micrographs of the specimens in heat treated conditions. It is observed that there are many globular particles on the surface of the specimens. The surface is optically smooth and of low porosity. No obvious surface damage is found. SEM micrograph of the worn surface after friction testing is presented in Fig. 4(b). It is clear from the figure that 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 microcutting and micro-loughing 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. From Fig. 5 (EDX spectra), it is found that coating contains 91.56% Ni and 8.44% P by weight. Fig. 6 shows the XRD plots of EN coating in as deposited and heat treated conditions. The XRD patterns in as deposited condition of microcrystalline peaks. But with heat treatment at 400^{o}C for 1 h, broad peaks of Ni_2P, Ni_3P and Ni_5P_4 are observed.

a

b

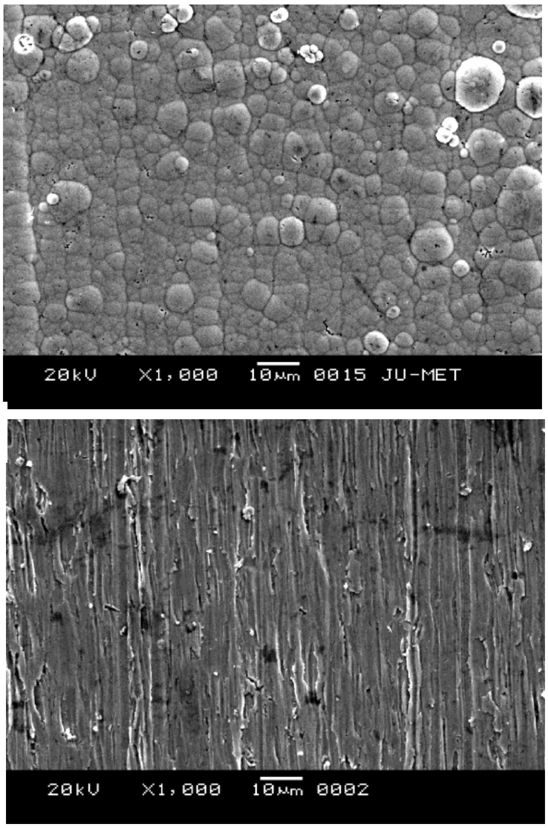

Fig. 4 SEM picture of EN coating (a) coated surface and (b) worn surface

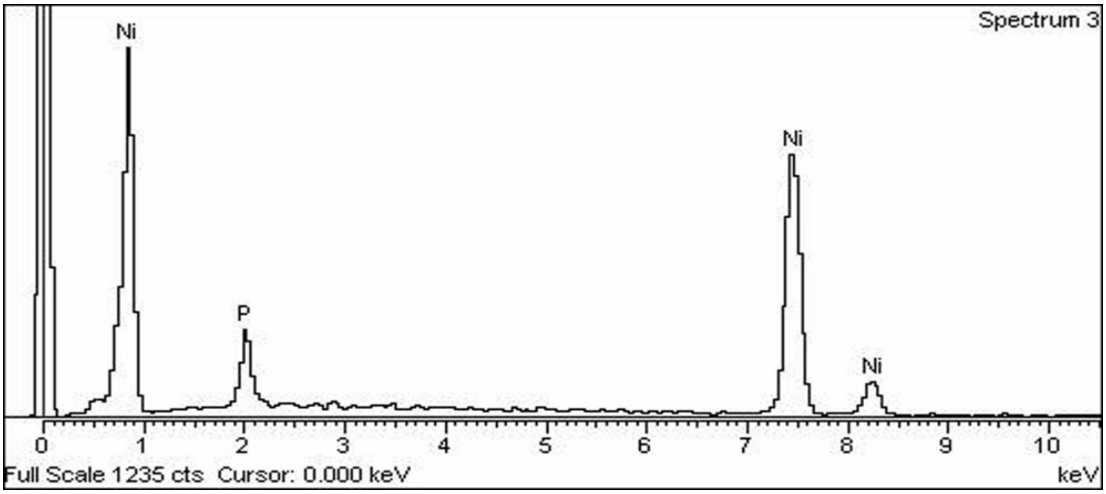

Fig.5 EDX spectra of EN coating.

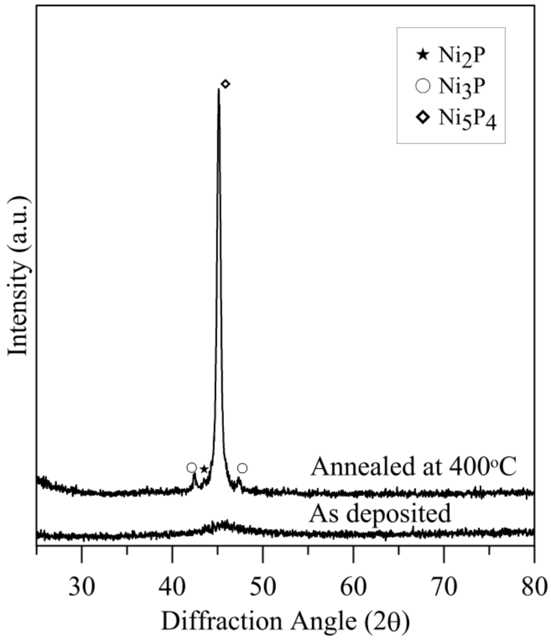

Fig. 6 XRD plot of EN coating

5. Conclusion

In the present study, Taguchi method is employed to optimize the coating process parameter to minimize friction of EN coatings in alkaline medium against rotating alumina disk. It is observed that bath temperature has the maximum contribution on friction coefficient. The interaction of reducing agent concentration bath temperature has the highest contribution in controlling frictional force of the EN coating. The optimal coating process parameter combination for minimum friction is A3B3C1D1 i.e, highest levels of nickel source concentration and reducing agent concentration along with lower level of bath temperature and annealing temperature. The friction coefficient is reduced by about 67% from initial to optimal. The microstructural study through SEM micrographs reveals that surface of the coatings is smooth of low porosity and dense with nickel and phosphorous content of around 91.5% and 8.5% respectively. In as deposited condition the coating is a mixture of amorphous and microcrystalline structure which generally turns crystalline with heat treatment. This is ascertained by the presence of Ni_2P, Ni_3P and Ni_5P_4 peaks in the XRD plot of Ni-P coating annealed at 400^oC.

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