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Optimization of microhardness of electroless Ni-P coatings using Taguchi technique

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ABSTRACT : *The present paper deals with the synthesis of chemically deposited nickel-phosphorous coatings on mild steel substrate and optimization of the hardness behaviour of the coating. There are several parameters associated with this coating procedure which can affect the hardness of the coating. In this study three coating process parameters are varied within a specific range and various thermal heat-treatment temperatures from 300°C to 500°C were also performed on the coating to obtain the maximum hardness. Thus it is a maximization problem which has been solved using Taguchi L_{27} orthogonal array design method. ANOVA is performed to determine the individual significance of parameters and finally a confirmation test is carried out to validate the result.*

Keywords: *Electroless Ni-P coating, microhardness, optimization, Taguchi method, heat treatment.*

I. INTRODUCTION

Recently, electroless Ni-P alloy coating has become popular in both scientific and industrial domains [1, 2]. Electroless nickel (EN) coatings have found numerous modifications to meet the challenging needs of a variety of industrial applications since its inception by Brenner and Riddell in 1946 [3]. Electroless nickel coatings are used in different areas such as aerospace, aviation, automotives, oil and gas processing, food processing, microelectronics, radio electronics, computer engineering, chemical processing, textiles, machinery, mining and metallization of plastic etc [4]. The hardness of Ni-P coating is mainly dependent on temperature of heat treatment. Heat treatment causes significant changes in properties and structure. The hardness and wear resistance of EN deposits can be increased by heat treatment due to the formation of nickel phosphide (Ni_3P) [5, 6]. Maximum Ni_3P is formed when annealed at 400^{o}C for 1 hour [7]. Hardness also increases with increase of coating thickness which is controlled by deposition time because electroless Ni-P deposition is a nucleation and growth phenomena which are time dependent [8]. Many researchers reported that the maximum hardness is obtained after a heat treatment at 400^{o}C for one hour [9] because the recrystallization temperature of Ni-P is 340^{o}C [10]. The present study investigates dependence of hardness characteristics of coating and optimization of coating parameter for maximum hardness based on Taguchi technique. The microstructure, composition and phase structure of the coatings are studied with the help of scanning electron microscopy (SEM), energy dispersive X-ray (EDX) and X-ray diffraction (XRD) analysis.

Taguchi design technique

Taguchi method [11] is used to optimize the electroless Ni-P coating parameters for maximum microhardness based on L_{27} orthogonal arrays (OA). Thus both time and cost of experimentation is decreased. Taguchi method uses S/N (Signal/Noise) ratio to identify the quality characteristics. Based on the criteria of the experiment, S/N ratio can be categorized as: lower the better (LB), higher the better (HB) and Nominal the best (NB). In the present study, microhardness needs to be maximized, thus higher the better criteria is used. Furthermore, a statistical analysis of variance (ANOVA) is performed. With the use of both S/N ratio and ANOVA analysis, the optimal combination of coating parameters is predicted.

II. DETAILS OF EXPERIMENT

2.1 Deposition of coating

The Ni-P coating is deposited on mild steel (AISI 1040) substrate of size 4 mm dia × 30 mm long. The substrates, before coating are subjected to pickling treatment in dilute (50%) hydrochloric acid for one minute to ensure the removal of surface layer formed like rust or other oxides. Subsequently, the samples are rinsed in distilled water followed by methanol cleaning prior to coating. The selected bath composition and operating condition of electroless Ni-P coatings is reported in [4]. EN deposition is carried out 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°C temperature and placed in the bath for deposition. The coating is carried out for a period of 4 hours. 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 is over, the samples are washed with distilled water. The coated samples are annealed in a box furnace for 1 hour at different temperatures (300°C, 400°C, and 500°C) according to the L_{27} OA.

2.2 Design of experiment

Four coating process parameters namely, concentration of source of nickel (A), concentration of reducing agent (B), deposition temperature (C), and annealing temperature (D) are considered. A has levels 30, 40 and 50 g/l, B has levels 10, 17 and 24 g/l, C has levels 80°C, 85°C and 90°C while factor D has levels 300°C, 400°C and 500°C. All these levels are marked level 1, 2 and 3 respectively in ascending order. It is a four-factor three-level experiment. The total degree of freedom (DOF) considering the individual factors and their interaction is 20. Here, L_{27} OA is chosen 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, microhardness of Ni-P coating is taken as the response variable.

2.3 Measurement of microhardness

Hardness is the property of material which offers resistance to indentation or scratch. Hardness is generally characterized by strong intermolecular bonds. Hardness values offer a comparative measure of a material’s resistance to plastic deformation from a standard source, as different hardness techniques have different scales. The Vickers hardness test is a commonly used test due to its wide load range capability. Vickers hardness (HV) is calculated as:

$$HV = \frac{1.8544F}{d^2} \qquad (1)$$

Where load (F) is in Kilogram force and the mean of two diagonals created by the pyramidal indent (d) is in millimeter. Microhardness testing of Ni-P coatings is carried out in a UHL microhardness tester (VMHT MOT, Sl. No. 1002001, Technische Mikroskopie) with a Vickers diamond indenter. Microhardness testing of Ni-P coatings is employed by precise diamond indenter with 1 kgf load. The dwell time is kept at 10 s while the speed of indentation is set at 50 µm/s. It should be mentioned here that the hardness is measured just after the coating is over with or without heat treatment and no separate surface preparation is done. An average of at least three hardness values for each sample is reported in Table 1. It may also noted here that the indentation depth (h) is related to mean diagonal (d) by the relation, h = d/7. In the present experiment, the maximum value of d is found to be around 46 µm. Hence, h is evaluated as 46/7 = 6.57 µm. Thus the depth of indention is much smaller compared to the coating thickness. This implies that the measured value of hardness represents the hardness of the coating itself with no effect of the substrate material coming into play.

2.4 Surface morphological and compositional test

Surface morphology study of the coating is done by SEM (JEOL, JSM-6360) to analyze the microstructure of the deposited coatings before and after annealing at various temperatures. 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 phase structure is studied with the help of X-ray diffraction (XRD) analysis (Rigaku, Ultima III) so that the different precipitated phases both pre and post heat treatment are identified.

III. RESULTS AND DISCUSSION

3.1 Analysis of signal-to-noise ratio

Taguchi method employs S/N ratio 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 microhardness as the performance index. The average of S/N ratio of each level of the factors of A, B, C and D is given in Table 2 and total average value of S/N ratio of all the 27 experiments is also listed in this Table. It is found that process parameter D has the highest delta value (rank 1). Hence, annealing temperature has the maximum influence on hardness of electroless Ni-P coatings. Parameters A and C are also found to have some influence on the hardness. But parameter B has the least influence on the hardness of the coating. The same observation is found in the main effects plot shown in Fig. 1. The optimal combination is obtained as A1B2C2D2 for maximum hardness.

Table 1 Response table for mean S/N ratio

Level	A	B	C	D
1	56.91	56.75	56.98	56.20
2	56.87	56.86	57.05	57.74
3	56.54	56.71	56.29	56.40
Rank	3	4	2	1
Delta	0.37	0.15	0.76	1.54
Total mean S/N ratio = 56.775				

Table 2 Results of confirmation test

	Initial condition	Optimal condition	
		Prediction	Experimentation
Level	A2B2C2D2		A1B2C2D2
Micro hardness (HV_1)	691		802
S/N ratio (dB)	56.7896	58.0150	58.0835

Improvement of S/N ratio = 1.2939 dB

3.2 Analysis of variance (ANOVA)

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 ANOVA (details are omitted for brevity), it is observed that parameter D has the most significant effect with confidence level of 95%. Parameter C is also significant but at a confidence level of 65%. Among the interactions, A×C has moderate contribution.

3.3 Confirmation test

After the optimal level of testing parameters have been found, it is necessary that verification test is carried out in order to evaluate the accuracy of the analysis and to validate the experimental results. Table 3 shows the comparison of the estimated S/N ratio with the actual S/N ratio using the optimal parameters. The improvement of S/N ratio from initial to optimal condition is 1.2939 dB, which means there is 16% increase in hardness from initial to optimal condition.

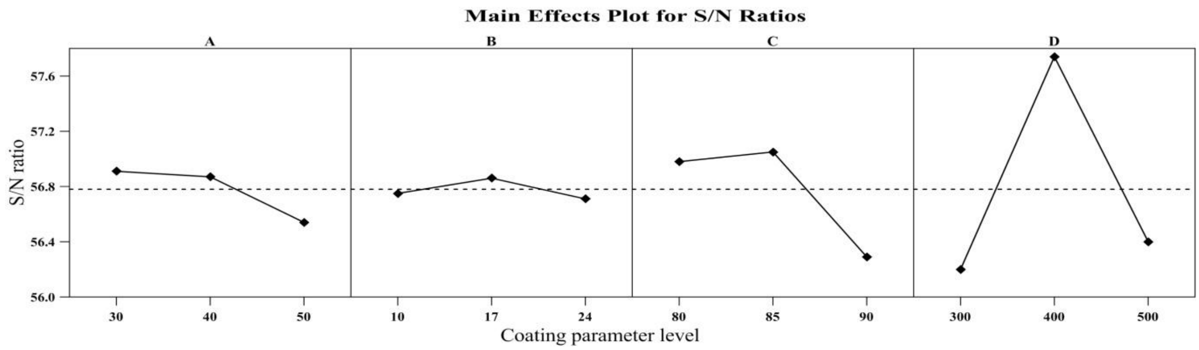

Figure 1 Main effects plot for signal-to-noise ratio

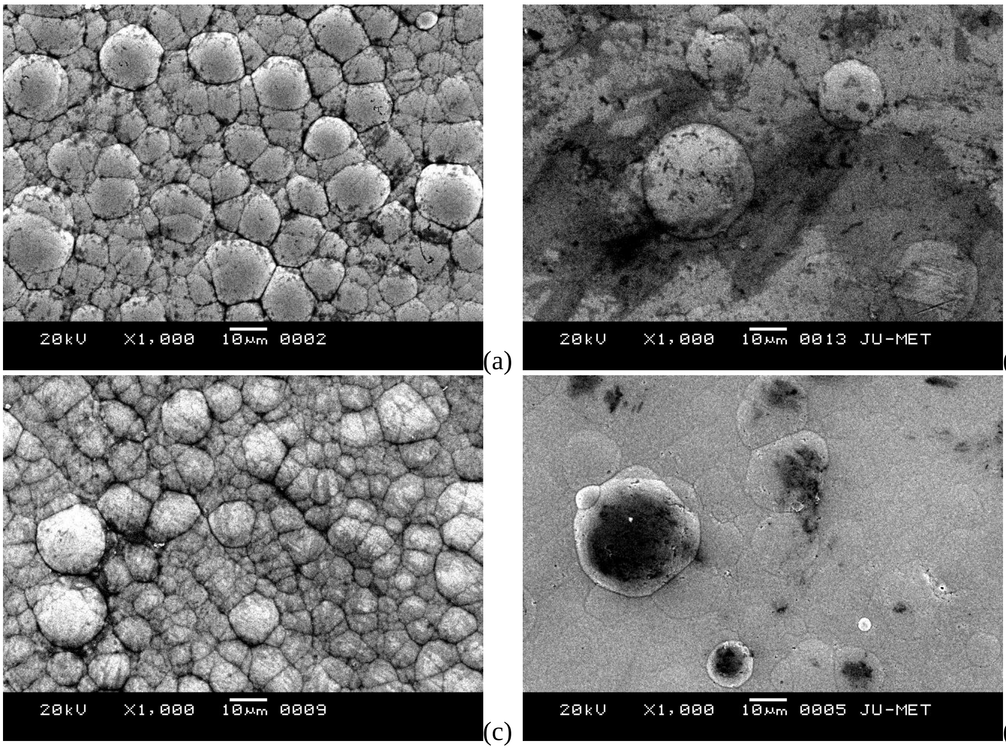

Figure 2 SEM picture of Ni-P coatings (a) as-deposited, (b) annealed at 300^{o}C, (c) annealed at 400^{o}C, and (d) annealed at 500^{o}C.

3.4 Surface morphology and composition study

The coated specimens as-deposited as well as heat-treated at different temperatures are studied by SEM. From Fig. 2 (SEM micrographs of the surfaces), it is seen that there are many globular particles on the surface of the substrate. The surface is optically smooth and of low porosity. No obvious surface damage is found. The surface of the Ni–P coatings appears to be dense. Also by careful observation, it can be noted that the Ni-P nodules are quite deflated and flat in as deposited condition but gradually grow in size with increase in heat treatment temperature giving rise to coarse grained structure. From Fig. 3 (EDX spectra), it is found that coating contains 91.56% Ni and 8.44% P by weight. Fig. 4 shows the XRD plots of as deposited and heat treated conditions. From this figure, it is evident that in as-deposited condition the phase is mostly amorphous but turns to crystalline with heat treatment because various nickel phosphide compounds (Ni_2P, Ni_3P and Ni_5P_2) are formed in heat treated specimen.

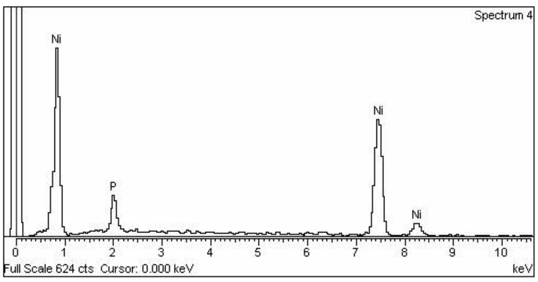

Figure 3 EDX spectra of Ni-P coatings

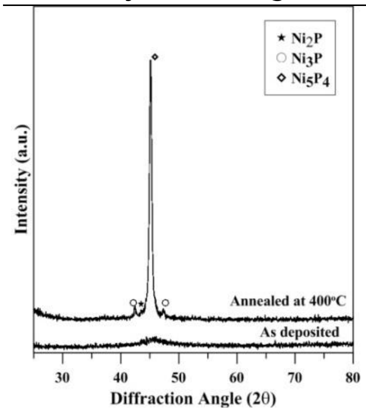

Figure 4 XRD plots for Ni-P coatin

IV. CONCLUSION

Taguchi method is used to optimize the coating process parameters to maximize the hardness of electroless Ni-P coatings. It is seen that annealing temperature has the most significant influence on hardness. The optimal coating parameter combination for maximum hardness is obtained as A1B2C2D2 i.e., lower level of nickel source concentration along with medium levels of reducing agent concentration, bath temperature and annealing temperature. The hardness is increased by about 16% from initial to optimal. It is observed 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. The coatings are amorphous in as-deposited condition but after the heat treatment, the coatings turn crystalline with different nickel phosphide compounds.

V. ACKNOWLEDGEMENT

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REFERENCES

[1] R. C. Agarwala, and V. Agarwala, Electroless alloy/composite coatings: A review, Sadhana, 28, 2003, 475-493.
[2] P. Sahoo, and S. K. Das, Tribology of electroless nickel coating – A review, Materials and Design, 32, 2011, 1760-1775.
[3] A. Brenner, and G.E. Riddell, Nickel plating by chemical reduction, US Patent US2532282, 1950.
[4] P. Sahoo, Optimization of electroless Ni-P coatings based on multiple roughness characteristics, Surface and Interface Analysis, 40, 2008, 1552-1561.
[5] K.G. Keong, W. Sha, and S. Malinov, Crystallisation kinetics and phase transformation behaviour of electroless nickel–phosphorus deposits with high phosphorus content, Journal of Alloys and Compounds, 334, 2002, 192-199.
[6] J.N. Balaraju, T.S.N. Sankara Narayanan, and S.K. Seshadri, Electroless Ni-P composite coatings, Journal of Applied Electrochemistry, 33, 2003, 807-816.
[7] D. Vojtech, M. Novak, M. Zelinkova, P. Novak, A. Michalcova, and T. Fabian, Structural evolution of electroless Ni–P coating on Al–12 wt.% Si alloy during heat treatment at high temperatures, Applied Surface Science, 255, 2009, 3745-3751.
[8] N. Sridhar, and K. Udaya Bhat, Effect of deposition time on the morphological features and corrosion resistance of electroless Ni-High P coatings on aluminium, Journal of Materials, Article ID 985763, 2013.
[9] H. Ashassi-Sorkhabi, and S.H. Rafizadeh, Effect of coating time and heat treatment on structures and corrosion characteristics of electroless Ni-P alloy deposits, Surface and Coatings Technology, 176, 2004, 318-326.
[10] D. Nava, C.E. Davalos, A. Martinez-Hernandez, F. Manriquez, Y. Meas, R.Ortega-Borges, J.J. Perez-Bueno, and G. Trejo, Effects of heat treatment on the tribological and corrosion properties of electrodeposited Ni-P alloys, International Journal of Electrochemical Science, 8, 2013, 2670-2681.
[11] P. J. Ross, Taguchi techniques for quality engineering (McGraw-Hill, New York, 1996).