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Optimization of process parameters to improve the wear resistance of the mild steel using electroless Ni-P coating process

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KEYWORDS

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SEM
XRD
Wear
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ABSTRACT

The electroless coating has several advantages over other coating techniques available in the market. They are the formation of a uniform deposit on irregular surfaces, direct deposition on conductors and surface activated non – conductors, formation of less porous in the deposits, very good wear resistance and corrosion resistance. In addition, it has few drawbacks of EN coating are the cost of chemicals used are high, poor nickel recovery from the bath, electroless nickel bath decomposition. Due to these reasons, the EN coating process efficiency is reported to be poor and is of the order of only 50%. Hence it is felt that a detailed investigation on the possibility of improving the coating efficiency and wear behaviour of electroless coatings by optimization techniques. The main objective of this work is to investigate the process parameters influencing the wear behaviour of the electroless Ni-P coating process. The result showed that after optimization of process parameters, the confirmation test is conducted and found a suitable combination of parameters are identified to reduce the wear rate. The wear resistance of the mild steel substrate is improved by 65 % when compared to the uncoated substrate.

1.0 INTRODUCTION

Electroless plating works without electricity to reduce the ions into metal using a reducing agent. After the involvement of technology in electroless plating, the efficiency is very high and according to the needs of application the thickness, colour, properties of the coating can be altered

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(Chandrasekar, 2021). The electrolyte can withstand up to 95 degree Celsius and thickness of 100 microns are possible without any pores in the deposit. The electroless coating has several advantages over other coating methods. The bright coating on the deposit is obtained which gives a good aesthetic appearance to the product (Muraliraja & Elansezhian, 2015). The surface finish is very smooth thus, it follows an autocatalytic process. The aid of electricity is not required for the process hence the deep holes, through holes, blind sites, and internal parts of any engineering components can be protected very easily from corrosion (Rosas-Laverde et al., 2020).

The wear resistance can be improved significantly by adopting electroless composite coating over the substrate (Ru et al., 2017). Tamilarasan et. al. improved the corrosion resistance of the coating by incorporating nano TiO_2 besides added two different surfactants into the electrolyte during the coating process. In analysis, it is reported that significant improvement in corrosion resistance of the material after electroless coating (Tamilarasan et al., 2015). Recent studies reported that the nickel recovery efficiency is improved by incorporating Zwitterionic surfactant into the electrolyte (Muraliraja et al., 2016). The wear resistance of the material is improved by adding several hard particles during the coating process one of the particles is called graphene oxide (Tamilarasan et al., 2017). In the same way, many hard ceramic particles are incorporated and improved the wear resistance considerably. Optimization is the method or technique to improve the efficiency of particular output by altering the input process parameters, which are variable (He et al., 2014). Among available methods, Taguchi is efficient and robust in design. Based on the orthogonal array the experiment are designed and conducted then the confirmation test is conducted to verify the output. Without the aid of the optimization process, the researchers tried to improve the wear resistance by incorporating the hard ceramic particles and metallic reinforcements. The coating can be done on the substrate in two categories they are amorphous and crystalline structure. During coating, at elevated temperature, the SiC particles react with Ni-P deposit, co-deposited, and form composite coating on the substrate. the results of the composite coating revealed that the wear resistance and hardness of the deposit improved significantly (Ma et al., 2014). Ni-P-TiN coating is made on the steel substrate to incorporate in the piston ring application. The coating improved the properties such as friction and weight loss of the ring due to wear (Xu et al., 2019). To improve the interfacial bonding between the particles the electroless coating is adopted thus without the help of chemicals or third materials the bonding between the materials are improved significantly (Fan et al., 2018). The biodiesel container is widely used everywhere to reduced the emission released from vehicles. But, the wall of the storage tank of biodiesel itself react with biodiesel and increases the release of NOx. Hence the electroless coating is preferred to stabilize the reaction and improves the corrosion resistance of the container (Sukkasi et al., 2011). The applications of the electroless coating has no limits which can be extended as required.

In this research work, the L9 Taguchi optimization is adopted to improve the wear resistance of the deposit (Nelabhotla et al., 2016). The coating is tested for surface morphology using a scanning electron microscope and elements are identified using energy dispersive X-Ray analysis. The grain formations are analysed using an X-Ray diffraction machine. The pin on the disc machine is used to test the wear resistance of the material.

2.0 EXPERIMENTAL PROCEDURE

The substrate is prepared for the required dimension of 9 mm diameter and 30 mm height bullet shape. The substrates are heat-treated at 800 degrees Celsius for 2 hours using a muffle furnace and furnace cooled to obtain a good crystal arrangement. To make the surface smooth the finishing operations such as grinding and disc polishing are done on the substrates. The surface finishing of the uncoated substrate is measured as 0.57 μm using a hand-held surface roughness tester. Before the coating process, surface activation is required to deposit the metal particles. The pre-treatment process for mild steel substrate is cleaning the substrate with acetone, acid pickling [8% $H_2 SO_4$ by volume] for 1 minute, cleaning with methanol. At last, the substrate is dipped into the electrolyte bath. The electrolyte used for the coating process should contain a source of nickel, reducing agent, stabilizer and complexing agent. The composition of the electrolyte and coating parameters used in the coating process are given in Table 1.

Table 1: Electrolyte composition for Ni-P coating.

BATH COMPOSITION	QUANTITY IN g/L
Nickel chloride	30
Sodium hypophosphite	40
Tri Sodium citrate	25
Sodium chloride	50
Surfactant (SLS)	1.2

The pH of the bath was maintained by the addition of a sufficient quantity of ammonia solution and maintained at 9. The electrolyte was heated indirectly by an electrically heated oil bath whose temperature was regulated by a Proportional Integral Derivative (PID) controller at 90 degrees Celsius. The temperature of the oil medium was controlled and the corresponding temperature of the electrolyte was monitored using a thermometer. The coating thickness T (mm) was obtained using the following formula (Alsari *et al.,* 2001):

$$T = \frac{W1-W2}{\rho \mathrm{AT}} \tag{1}$$

where W_1, W_2 are weights of the substrate before and after coating, (mg), ρ is density (gm/cm^3) and A is surface area exposed for coating (cm^2) and T is deposition time (hour). In this investigation, the coating thickness was determined by the weight gain method. Wear studies were performed on a Ducom pin-on-disc model TR-201 friction and wear monitor with a computer interfaced data acquisition system. The formula to estimate specific wear rate is given in the equation below.

$$Specific\ wear\ rate = \frac{W}{ALD} \tag{2}$$

To obtain a better recovery of nickel from the electrolyte, preliminary experiments were conducted and the levels are obtained as pH 5 and 9 in the bath. The input parameters such as levels and parameters are optimized. The selected parameters are pH, the concentration of RA, surfactant concentration and interval of adding an excess amount of RA. The 4 level design factors and in each factor 3 levels were considered and presented in Table 2. The benefit of the Taguchi technique is the required number of experiments are only nine and it is expressed as L1 to L9 and named as an orthogonal array as shown in Table 3.

Table 2 Process parameters selected and its levels.

Design factors	Unit	Levels		
		1	**2**	**3**
(A) Excess Reducing Agent	g/L	2	4	8
(B) Surfactant Concentration	g/L	1	1.2	1.4
(C) pH		5	7	9
(D) Adding Time	Minutes	30	60	90

Table 3: L9 orthogonal array developed using minitab software.

Experiment No.	Factors			
	A	**B**	**C**	**D**
1	2	1.0	5	30
2	2	1.2	7	60
3	2	1.4	9	90
4	4	1.0	7	90
5	4	1.2	9	30
6	4	1.4	5	60
7	8	1.0	9	60
8	8	1.2	5	90
9	8	1.4	7	30

3.0 RESULTS AND DISCUSSION

3.1 Influencing Factors and Their Rank

The main effect plot for mean and SN ratios are illustrated in Figures 1 & 2. This plot shows the effects of changing the parameters from one level to another. The horizontal line in the plot represents the overall mean of the experimental region, which is the average of the S/N ratio of the nine experimental matrixes (Kundu et. al., 2016). From the plot, it can be observed that parameters B and A have huge variations between the levels of each parameter thus it is understood that it has greater influence when compared to the coating thickness from the electroless bath. Parameters C are found to have very less influence over the coating thickness (He et al., 2014).

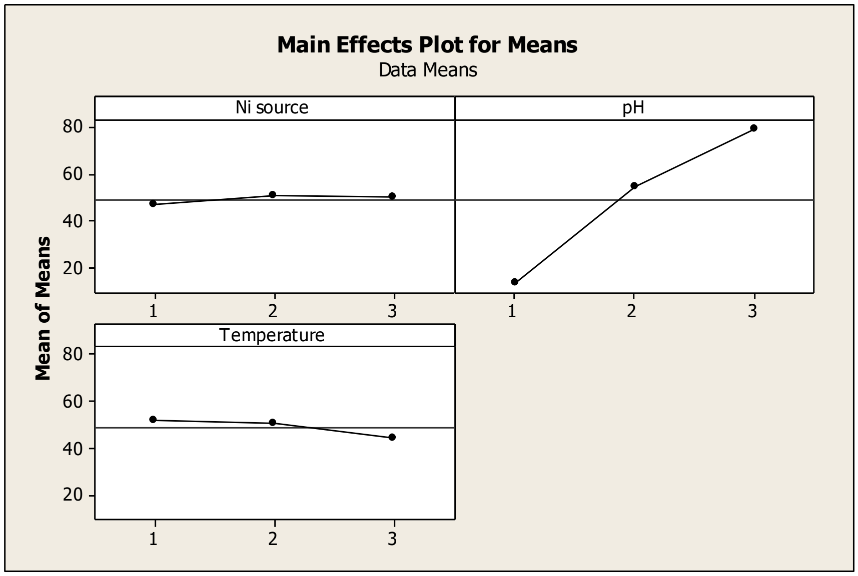

Figure 1: Main effects plot for mean.

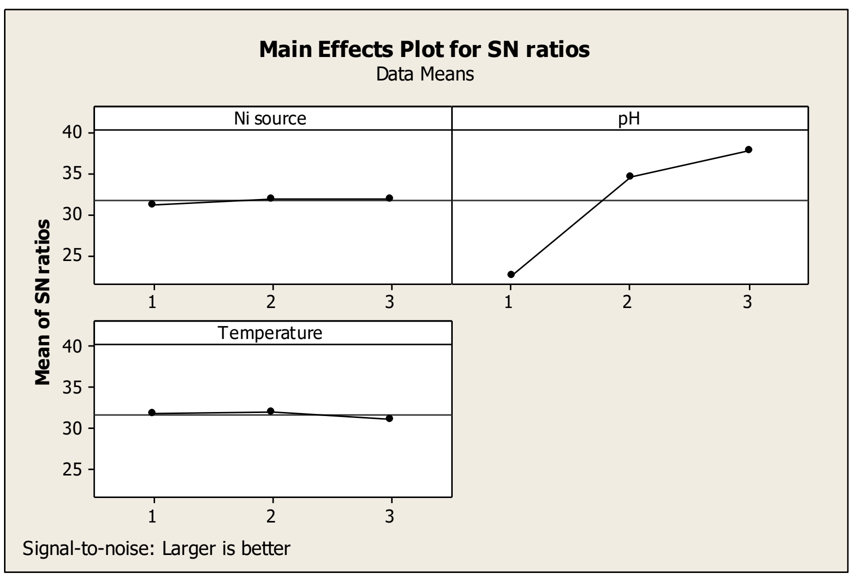

Figure 2: Main effects plot for SN ratio.

Figure 3 shows the interaction plots, there is no straight line of the plots are observed. Nonparallel lines are indicative of the presence of interaction, while intersecting lines are indicative of the presence of strong interaction. From the interaction plots (Figure 3), it is evident that lines intersect in all the plots, and hence, all the factors have some amount of interaction between each other (Muraliraja et al., 2014). Since the Taguchi method obtains the optimal level combination by choosing those levels, for which the S/N ratio is the larger, the better, the optimal combination of parameters is found to be A2B3C2. It is seen clearly from the main effects plot for SN ratio, the most influencing coating parameter are pH, nickel source and temperature.

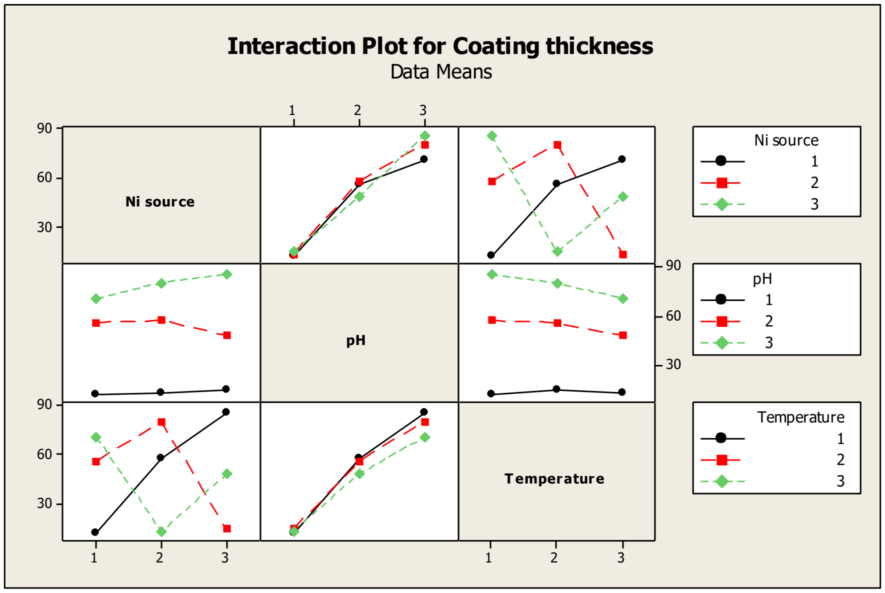

Figure 3: Interaction plot for coating thickness

3.2 Surface Morphology of Electroless NI-P Coatings

The morphology of the EN coatings produced as per design data in L9 was observed using the scanning electron microscope. The SEM micrographs of EN coatings are presented in figure 4. The coated surface shows bubble-like features distributed all over the substrate surface. These features are produced when the bases of hydrogen gas bubbles produced temporarily prevent direct contact between the electrolyte and the catalytic surface at the localized area concerned, while all around there will be a continued reaction (Sahoo & Das, 2011). Most uniformity in the microstructure was found in coating obtained from [Figure 4 (I)]. The non-uniformity of the surface was due to the different Ni % in the coating. The uniformity decreased with increasing bath pH value as depicted in Figures 4 (H) and 4 (D). it is a common understanding that the increase in nickel content in the deposit reduces the uniformity in the morphology of the deposit. i.e. with declining the phosphorus content in the deposit. Figures 4(A); 4 (C) and 4(G) shows bubble-like features distributed all over the substrate surface. The brightness of the deposit depends on the percentage of P content in the deposit. If the nickel content in the deposit is high then the deposit appears dual in the finish (Jagannatham et al., 2015).

3.3 EDX Analysis for Determination of Ni & P Content

The percentage of Ni and P content in the substrate was analysed using EDX diffractogram and the percentage of available concentration is shown in graph Figure 5. it is one of the evidence for improving the hardness value in the substrate. The electroless coating is a combination of nickel and phosphorous, Nickel will improve the hardness of the material obviously if hardness increases, the wear resistance of the material will improve. Another one is phosphorous especially for improving the corrosion behaviour; it is not for enhancing the hardness or wear. Hence, wear resistance will improve for trial 6 (optimized combination of parameters).

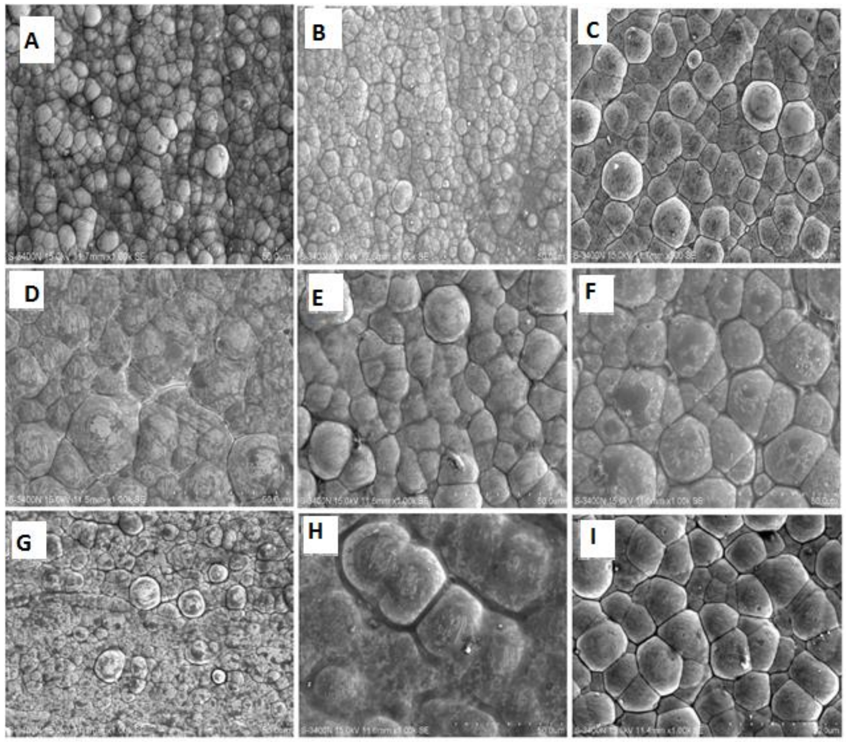

Figure 4: SEM micrographs (1000X) of EN coatings (a)Trial 1; (b) Trial 2; (c) Trial 7; (d) Trial 8; (e) Trial 5; (f) Trial 4; (g) Trial 3; (h) Trial 9; (i) Trial 6.

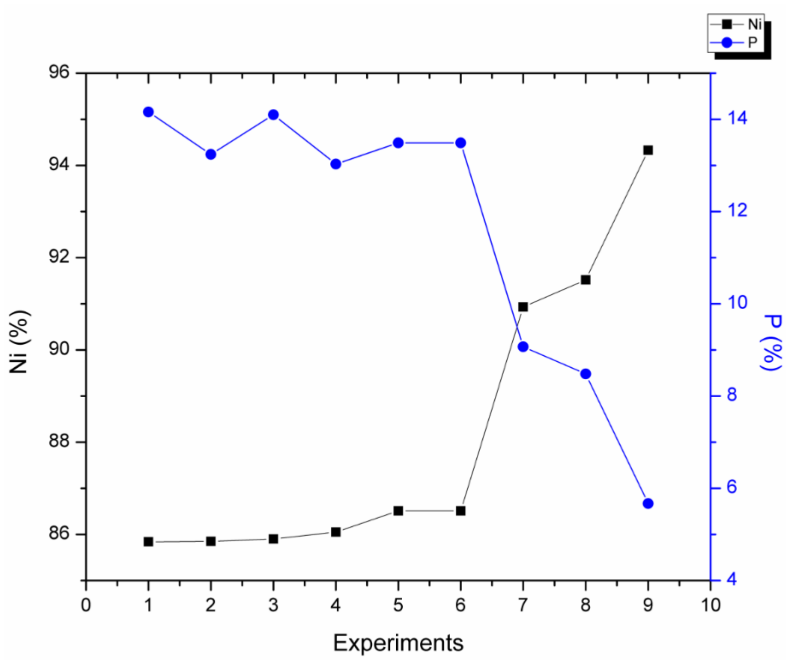

Figure 5: Presence of Ni and P % coated on the substrate.

3.4 X-Ray Diffraction Study

Typical diffractograms of samples coated as per the design data from the L9 orthogonal array are presented in figure 6. In all the cases, the range covers the (111) reflection of nickel occurring around 57.03^0 (2θ) along with the amorphous profile from the deposit which overlaps with the (111) reflection of nickel (Vitry & Bonin, 2017). The individual XRD profiles corresponding to the amorphous and crystalline phases were obtained through the deconvolution technique (Mahayuddin et. al, 2020).

3.5 Coating Thickness

In this investigation, the coating thickness was determined by the weight gain method. Using optimization techniques the experiment was conducted and it was observed that the maximum coating thickness was obtained at trail 5. The coating thickness and deposition rate in hours is shown in Figure 7.

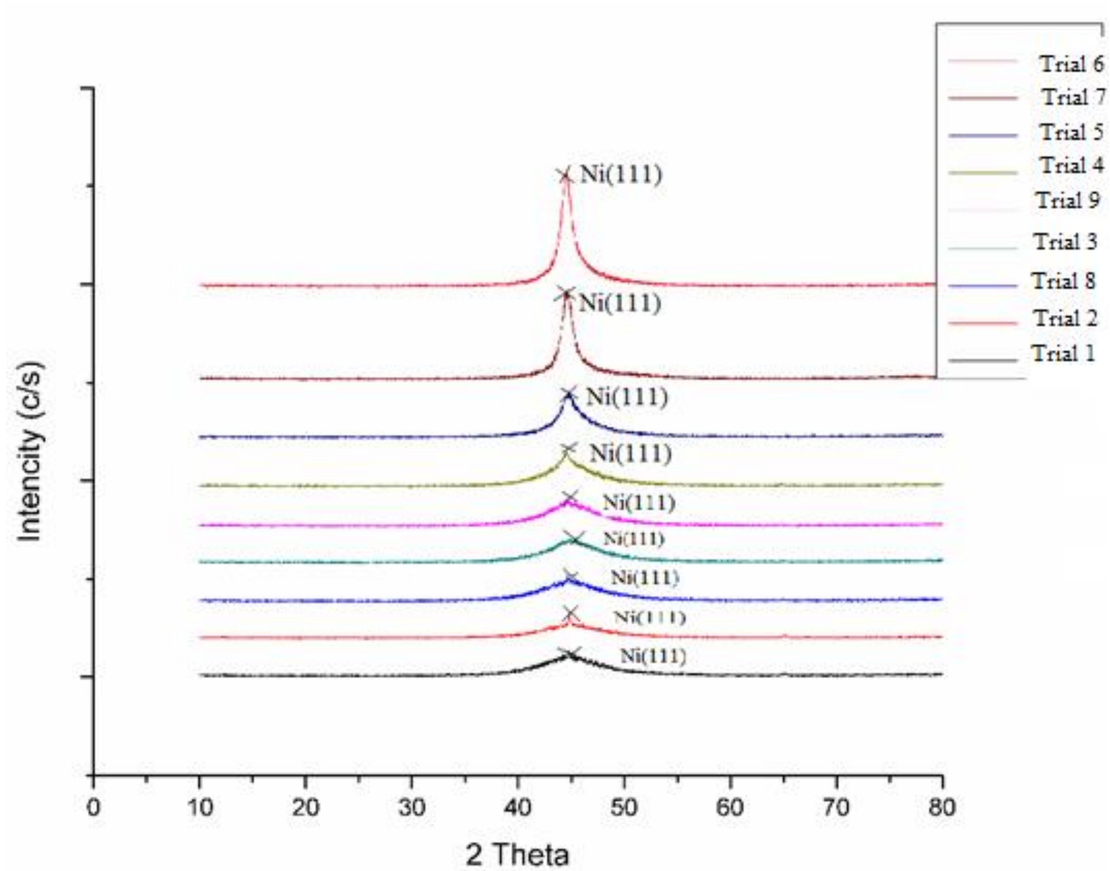

Figure 6: XRD Difractogram of all the substrates conducted using Taguchi techniques.

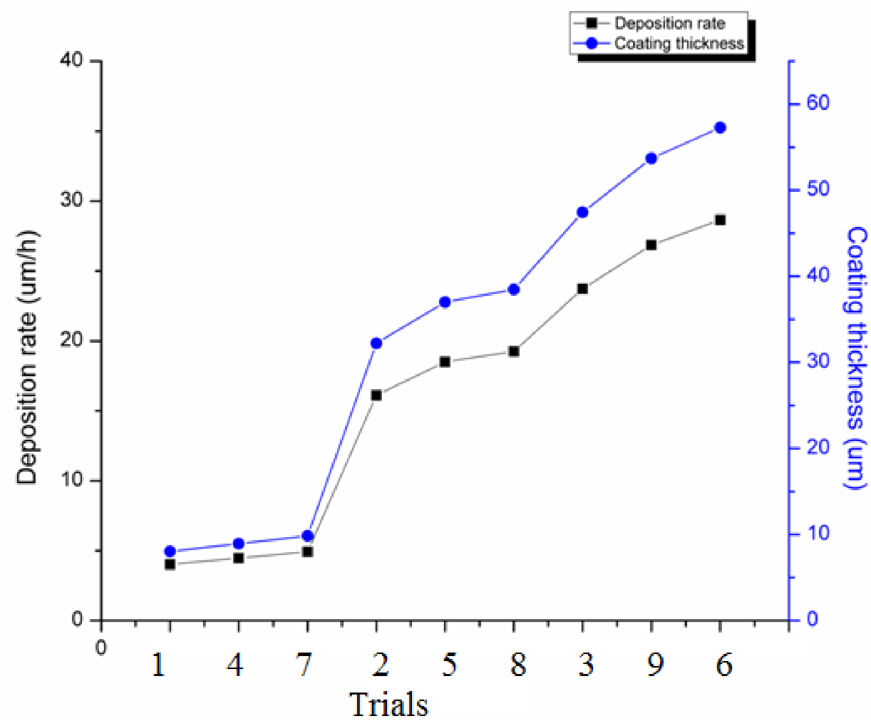

Figure 7: Graph for coating thickness and deposition rate.

3.6 Effect on Specific Wear Rate

The specific wear rate of the coated substrates is shown in figure 8. It revealed clearly that with increasing surfactant concentration, the deposit showed better resistance to wear (Ajibola, 2016). To understand the mechanism of wear in the coated substrate, the wear tracks of substrate were studied using SEM micrographs shown in figure 9. The coating developed without the addition of excess reducing agent got severe damage and the possible reason could be poor adhesion between the deposit and the surface of the substrate, low hardness of the deposit and ductility. The addition of surfactant in the electroless bath supports the formation of debris involvement to an improvement in the contact area between tribological pairs (Arulraj & Palani, 2018). Patchy removal of materials is observed in the SEM morphological analysis for the substrate produced without the addition of surfactant. The torn patches and detachment of deposit in few places were observed for the coating deposited with the addition of surfactant which cleary states the improvement of adhesion (Sudagar et al., 2013)(Kamdani et al., 2019).

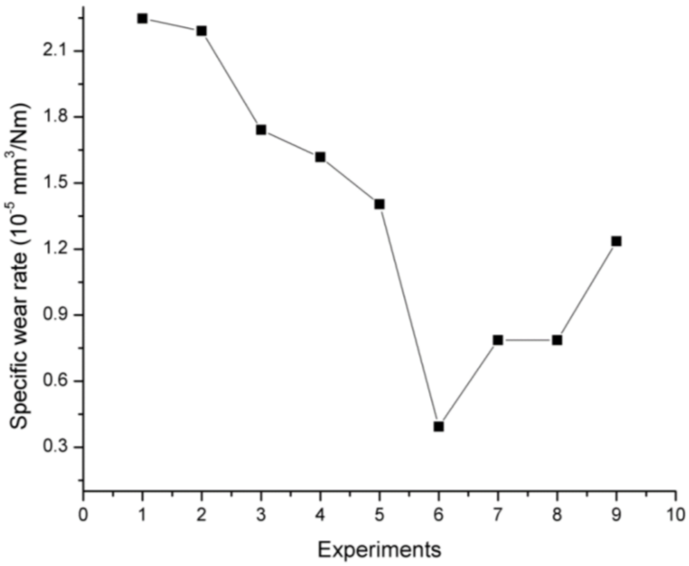

Figure 8: Specific wear rate of all the conducted experiments.

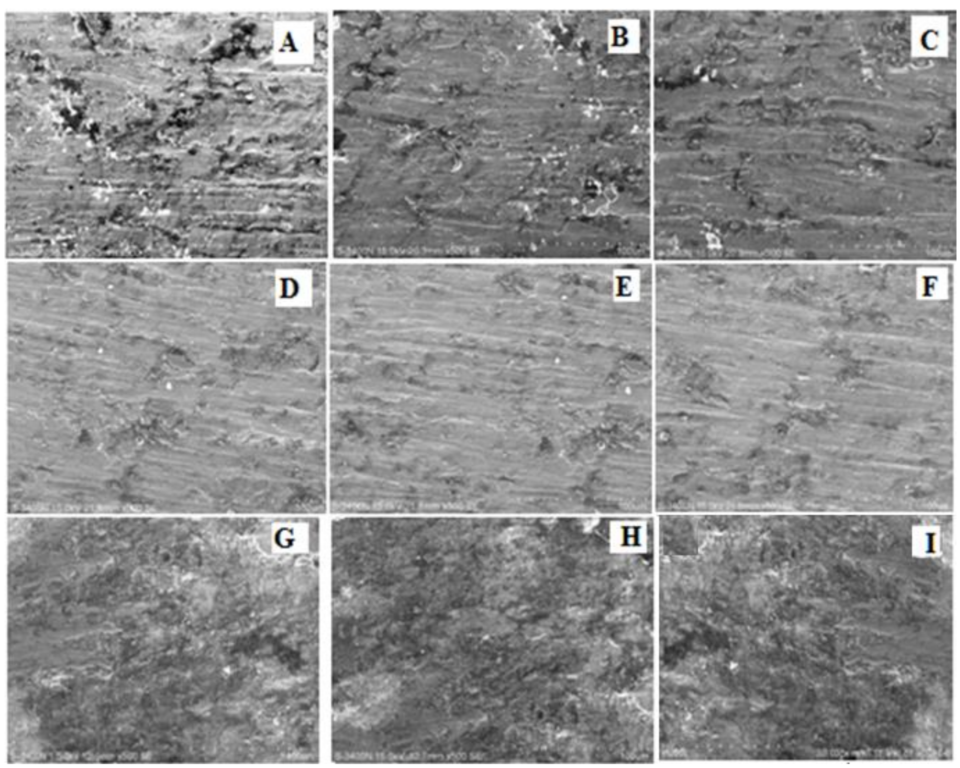

Figure 9: SEM micrograph for wear track of all experiments.

CONCLUSION

The Taguchi optimisation technique is successfully implemented in this work with an L9 orthogonal array for the process parameters of electroless coating namely Nickel source (A), pH (B), and Temperature (C). The optimized form to improve the wear resistance is derived to be $A1B2C2D2$. The parameters pH and temperature are significantly improving the deposit thickness and 58.2% more nickel was recovered from the electrolyte bath from the existing one. A moderate amount of influence is found to exist among all the factors. The phosphorus content of the coating is found to lie at 5.67%. The XRD plots showed that the electroless Ni-P coating is a mixture of amorphous and crystalline phases in as-deposited conditions but in our result, the crystalline phase is dominated so mechanical properties improved because of the presence of more amount of nickel present in the substrate. Nickel recovery efficiency is increased from 25 to 61%. Coating thickness is increased from 40 to 59 μm i.e. 32% improved from the conventional coating process. The specific wear rate is improved by 65% when it slides over a hard surface.

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