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Experimental Investigation on the Effects of Electroless Nickel Phosphorus Deposition, pH and Temperature with the Varying Coating Bath Parameters on Impact Energy by Taguchi Method

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Abstract**—**This paper discusses the effects of sodium hypophosphite concentration, pH, and temperature on deposition rate. This paper also discusses the evaluation of coating strength, surface, and subsurface by varying the bath parameters, percentage of phosphate, plating temperature, and pH of the plating solution. Taguchi technique has been used for the analysis. In the experiment, nickel chloride which is a source of nickel when mixed with sodium hypophosphite has been used as the reducing agent and the source of phosphate and sodium hydroxide has been used to vary the pH of the coating bath. The coated samples are tested for impact energy by conducting impact test. Finally, the effects of coating bath parameters on the impact energy absorbed have been plotted, and analysis has been carried out. Further, percentage contribution of coating bath parameters using Design of Experiments approach (DOE) has been analysed. Finally, it can be concluded that the bath parameters of the Ni-P coating will certainly influence on the strength of the specimen.

Keywords**—**Bath parameters, coatings, design of experiment, fracture toughness, impact strength.

I. Introduction

ELECTROLESS plating is a surface engineering process which involves deposition of a metal-metalloid alloy coating on various substrates. Karibasavaraja [11] has opined that electroless nickel coating has received widespread acceptance as it provides high hardness and excellent resistance to abrasion and corrosion wear. Electroless nickel coatings are used for the application of corrosion protection in a variety of environments. It is a barrier coating which protects the substrate from the corrosive environments by sacrificial action. However, in this respect, only electroless Ni–high P coating has been identified as an effective coating and extends excellent protection. Electroless Ni–Low P and Ni–medium P coatings have not been recommended for severe environments. Due to its properties of resistance to corrosion and high hot hardness, the process has wide application on items in the use of pump, valves, parts and is used to improve the life of components. With the appropriate use of pre-treatment and precise process control better service and adhesive performance can be obtained with the electroless nickel coating deposited on a multitude of metallic and non-metallic substrates [11]. The properties of electroless nickel coatings depend largely on the composition of the bath used and the deposition conditions. The small amounts of additives are usually used to provide improved deposition rate, throwing power and brightness, finer grain structure, and better corrosion resistance. Since electroless nickel plating process is chemical reduction of nickel ions to the nickel results from the presence of reducing agent in the solution, it always concerns with hydrogen evolution during the plating process and formation of porous surface. It greatly reduces the corrosion resistance of the coating. To remove this hydrogen from the surface of substrate and to produce pit free nickel deposits, surfactants were used as wetting agent into the plating. Pit free nickel deposits had been achieved by the addition of 150 ppm sodium dodecyl sulphate in the plating bath. The cleaning, pretreatment, and the coating bath parameters play a vital role in the effectiveness of the coated surface.

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II. Literature Survey

Dan et al. [1] have discussed the difference between two families of deposition techniques CVD and PVD and its influences on the choice of substrates and properties of the coating substrate systems. Lee Hyland [2] has opined that electroless nickel prepared using sodium hypophosphite as redundant and coatings which contain phosphorus are superior compared to those of the pure metal. Agarwala and Agarwala [3] have reviewed different electroless alloy/composite coatings with respect to its bath types, compositions, properties and its applications. They have carried out different characterisation studies on various electroless nickel-based coatings with emphasis on wear and corrosion properties. Narayanan et al. [4] have conducted experiment on Ni–P/Ni–B duplex coatings by electroless plating process and carried out evaluation of hardness, wear resistance, and corrosion resistance. They [4] have compared microhardness, wear resistance, and corrosion resistance of electroless nickel duplex coatings with electroless Ni–P and Ni–B coatings of similar thickness. Ramaseshan et al. [5] have concluded that titanium and aluminide intermetallic coatings are gaining greater importance due to their high melting temperature, low density, high temperature properties, and oxidation resistance. They also opined that vapour phase deposition methods can be used for coating low based carbon fibres. Hari Krishnan et al. [6] have discussed the development of electroless Ni-P bath their advantages, mechanisms of deposition, and applications. Mayer [7] has researched on trends of electroless nickel plating and concluded that electroless nickel deposits are found on functional parts such as office equipment spacers to state-of-the-art multi-chip module electronic packages. Molla et al. [8] have carried out an investigation to study the electroless nickel–phosphorus (Ni–P) deposition with high hardness and excellent resistance to wear and abrasion. In this study, autocatalytic deposition of Ni–P alloy has been carried out on steel CK-75 sheets from bath containing nickel sulphate hexahydrate, sodium hypophosphite hydrate, thiourea, lactic acid, and sodium acetate. Ayoub [9] had discussed the characteristics of electroless Ni-P plating treatment as applied to stainless steel substrate for improving its corrosion resistance and micro-hardness. Hino et al. [10] have conducted experiment to know the effects of alloying elements on zincate treatment and adhesion of electroless Ni-P coating onto various aluminum alloy substrates.

III. Experimental Work

The electroless Ni–P coating is made by using hypophosphite-reduced electroless nickel plating bath. Nickel chloride is taken as the source of nickel and sodium hypophosphite as the reducing agent.

A. Substrate Material

1. Medium Carbon Steel (C-40 steel)

The chemical compositions of the test material are depicted in Table I, and the physical properties of the substrate are depicted in Table II.

TABLE I
Chemical Compositions of Mild Steel

Content	C	Si	Mn	P	Pb	S	Fe
Volume %	0.14	0.05	0.90-1.30	0.11	0.20-0.35	0.27-0.33	average

TABLE II
Physical Properties of C-40

Property	Value
Hardness	170 HV
Yield Stress	270 N/mm^2 min
Tensile Strength	590 N/mm^2 max
Percentage of Elongation	30 min

2. Specimen Preparation

The coating specimens are prepared according to ASTM E23 standard. Coating of metals with electroless Ni-P coatings is highly dependent on substrate surface condition. The notch of 2 mm depth is made at the centre as shown in Fig. 1. The prepared samples are polished or grounded to get fine surface finish.

3. Coating Setup

In this experimental work, electroless nickel phosphorus coating has been done on the substrate by using the chemicals and appropriate percentage of ammonium chloride, nickel chloride, hydrochloric acid, sodium- hypophosphite, sulphuric acid, sodium hydroxide, and ammonium hydroxide. The composition of electroless nickel bath is depicted in Table III, and the setup is as shown in Fig. 2.

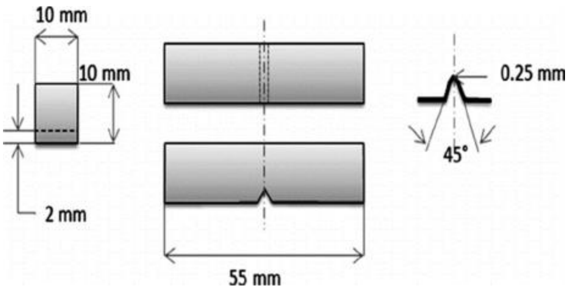

Fig. 1 Impact Specimen

TABLE III
Composition of Electroless Plating Bath and Plating Conditions

Bath Parameters	Value
Nickel chloride	30 g/L
Sodium hypophosphite	10 g/L
Ammonium chloride	50 g/L
pH	8-10
Temperature	70-90 °C
Plating time	60 mins


Fig. 2 Schematic diagram of coating setup

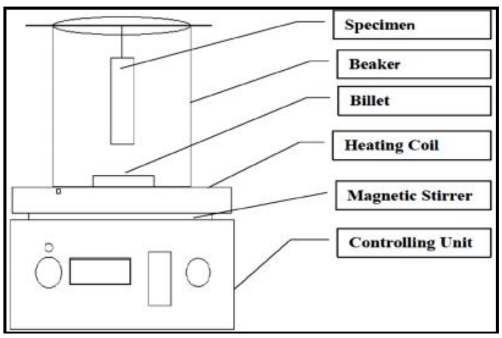

Fig. 3 Coating setup

4. Experimental Setup

An experimental set up is done by taking Mild Steel as the substrate material based on ASTM E23 standards. The standard setup of the arrangements is shown in Fig. 3. The specimen has been prepared by providing a notch at the centre of the specimen by taking the depth as 2 mm to record the deposition of Ni-P films. The chemicals are prepared based on the tabulated results indicated in Table IV and suitable coating bath has been prepared. The mixtures are heated to increase the bath temperature and subsequently its temperature is monitored. The samples are cleaned to remove the foreign particles and corrosion products prior its coating. The samples have been cleaned with deionised water. These specimens after thorough cleaning are subjected to pickling treatment with dilute hydrochloric acid for a minute in order to remove surface layer. Finally, the specimens are once again cleaned with deionised water prior to coating. The cleaned samples have been activated in sodium hydroxide solution. The specimens were again rinsed in deionised water in order to clean the acid which adhered on the surface of the specimen. The specimen is placed in the bath for deposition duration of 60 minutes. For each sample, the temperature of the plating solution and plating time has been recorded. The range of coating thickness was observed to be between 10-15 microns. After deposition, specimens have to be left to dry completely.

It has been observed that the pre-treatment process can significantly affect the corrosion resistance of electroless Ni-P coatings. These problems have been observed due to the improper electroless nickel plating and due to the failure to clean and pretreat surfaces.

5. Characterisation of Coated Specimen

Characterization of coating is done through impact test, which enables the influence of parametric variation on the coated specimen.

a. Impact Test

A low velocity pendulum type impact testing machine is used and it is illustrated in Figs. 4 and 5. The amount of energy absorbed to fracture the test piece has been measured.

In this experimental work, impact strength is evaluated by using a specimen with corner notch. It has been observed from the literature that corner notches are more prone to failure compared to other forms of notches. To validate the results, some modifications have been done to the notched specimen, and the results are analysed. The test specimen is shown in Fig. 5. Finally, the test specimen is placed in the impact testing machine with an inclination of 450.

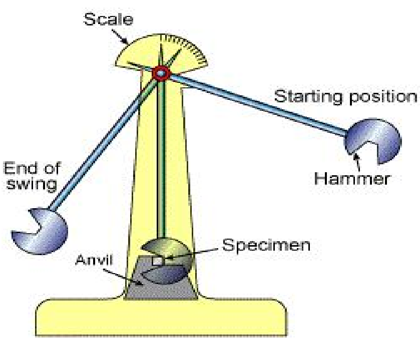

Fig. 4 Impact testing machine

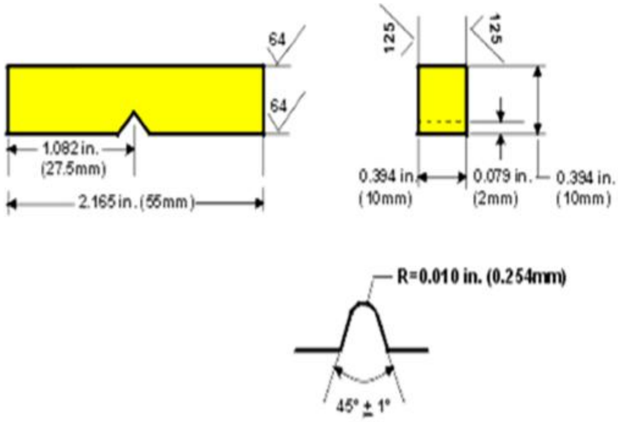

Fig. 5 Impact Test Specimen Specification

6. Design of Experiments (DOE)

DOE is based on the objective of desensitizing a product's performance characteristic(s) to variation in critical product and process design parameters. Through DOE, a series of tests are performed, where pre-planned changes are made to the controllable variables so that the reason for changes in the response can be observed and identified. The data collected from all the experiments in the set are analysed to determine the effect of various design parameters. The impact energy as a result of impact test was subjected to analysis of variance (ANOVA). The parameters chosen in the present work are given in Table IV.

TABLE IV
PARAMETERS IN DOE

Levels	pH	Temp.	Reducing Agent
1	6	70	10
2	8	80	20
3	9	90	30

In the present investigation, an L27 orthogonal array is chosen. This has 27 rows and 13 columns as shown in Table V.

The parameters chosen are pH, temperature, and reducing Agent. The experiments have been carried out on 27 tests and have been assigned with various parameters. Column I in table has been assigned to pH, column II has been assigned to temperature, column III has been assigned to the reducing agent. The remaining columns were assigned to appropriate columns based on their interactions.

IV. Results and Discussion

The experimental investigation on electroless nickel-phosphorus (Ni-P) coating, were made by using both acidic and alkaline hypophosphate reducer electroless plating. Nickel chloride was the source of nickel, whereas sodium hypophosphite served as the reducing agent and the source of phosphate and the sodium hydroxide were used to vary the pH of the coating bath. The experimental study was made by varying the bath parameters which includes the percentage or concentration of phosphate, plating temperature, and pH of the plating solution. The coated samples were tested for hardness and impact energy by conducting microhardness test and impact test. The surface roughness of the coated samples was analysed using surface roughness tester.

A. Effect of Coating Bath Parameter on the Impact Energy Absorbed

The different types of standard notches were prepared in the specimens to analyse the material properties. It has been observed that, there has been a stable crack growth except at the location of corner notch. This is due to more cleavage triggering sites found in specimen with corner notches. This cleavage triggering sites have caused unstable crack growth and lesser fracture toughness. Hence, it can be inferred that corner notched specimens are more prone to failure, and cleavage triggering sites have been the crack initiating points.

1. Effect of Temperature on Impact Energy Absorbed

In this study, the influence of electroless nickel phosphorus plating bath temperature and impact energy absorbed by the specimens was analysed. The impact energy for different parameters conditions was plotted and the contribution of temperature on the electroless nickel plated specimens and impact energy absorbed were analysed.

Fig. 6 represents temperature effect on impact energy absorbed by the electroless Ni-P coated samples. The obtained impact energy absorbed from the impact test for all the combination of parameters was plotted, and the contribution of temperature on the impact energy absorbed of the electroless nickel coated specimen was analysed. Fig. 7 shows the dependence of impact energy absorbed on the coated samples with the change in temperature of an electroless nickel phosphorus bath. It was observed that the impact energy absorbed increases with the increase in phosphate percentage at a temperature range of 70 °C. At 80 °C temperature, the impact energies absorbed for the 6 pH and 8 pH were more compared to 9 pH. Similarly, at 90 °C temperature, impact energies absorbed for the 6 pH and 8 pH absorbed seem to decrease.

TABLE V
L_{27} STANDARD ORTHOGONAL ARRAY

$L_{27}(3^3)$	1	2	3	4	5	6	7	8	9	10	11	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	2	1	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

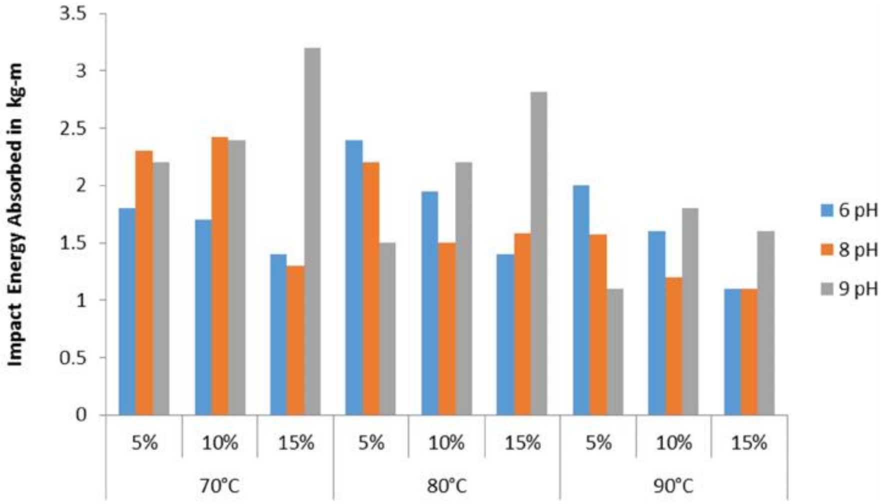

Fig. 6 Effect of temperature on impact energy absorbed

Fig. 7 represents pH effect on impact energy absorbed of the electroless Ni-P coated samples. The obtained impact energy absorbed from the impact test for all the combination of parameters was plotted, and the contribution of pH on the hardness of the electroless nickel coated specimen is analysed. The orthogonal array applied on electroless Ni-P coating experiment and the combination of parameters and the adsorbed energy have been recorded in each of the case. The parameters and levels chosen are given in Table VI, and the value of adsorbed energy for different parameters obtained is depicted in Table VII.

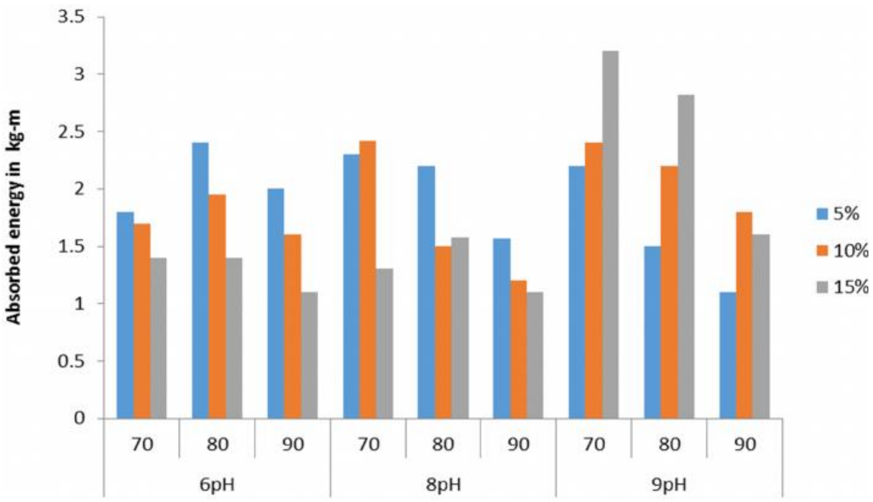

Fig. 7 Effect of pH on impact energy absorbed

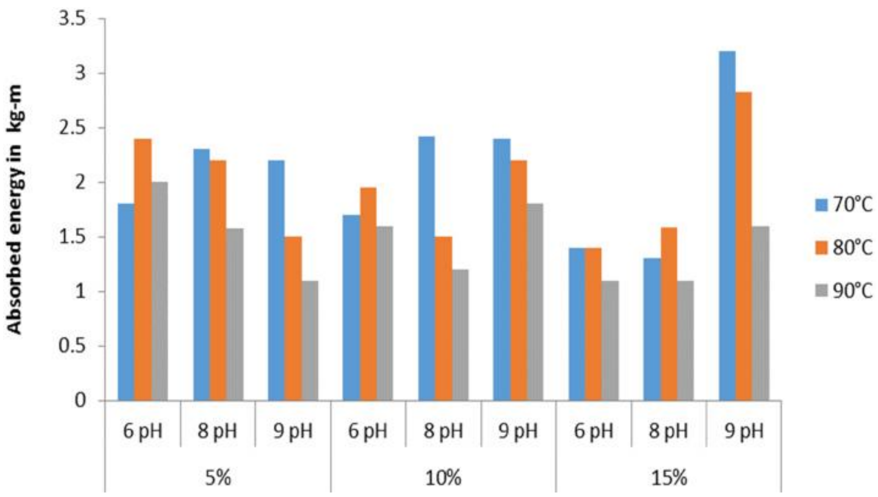

Fig. 8 Effect of phosphate content on impact energy absorbed

2. Effect of Phosphate on Impact Energy Absorbed

Fig. 8 represents phosphate effect on impact energy absorbed of the electroless Ni-P coated samples. The obtained impact energy absorbed values from the impact test for all the combination of parameters were plotted, and the contribution of phosphate on the impact energy absorbed of the electroless nickel coated specimen was analysed.

B. Determination of Percentage Contribution of Coating Bath Parameters by DOE

The experiments were conducted to plot the process parameters with their values at three levels, as shown in Table VI.

The experimental values have been transformed into S/N ratios by using a statistical Minitab software tool. The experimental results of impact test using L27 orthogonal array are depicted in Table VII.

TABLE VI
PROCESS PARAMETERS WITH THEIR VALUES AT THREE LEVELS

Levels	pH	Temp	Phosphate in grams
1	6	70^0c	05
2	8	80^0c	10
3	9	90^0c	15

1. DOE on Electroless Ni-P Coated Substrate

The study has been extended to know the characteristics of electroless Ni-P coating. Based on the principle of DOE, the experiments were reduced by incorporating the array table.

This array table consists of various permutations and combinations of parameters under consideration. For the analysis pH value, temperature, and reducing agent (phosphate) were taken as the parameters under consideration, and three levels of parameters were selected.

TABLE VII
EXPERIMENTAL RESULTS OF IMPACT TEST USING L_{27} ORTHOGONAL ARRAY

Sl. No.	pH	Temp In °C	Reducing Agent in grams	Absorbed Energy in kg-m	Impact Energy in kg $m^{1/2}$
1	6	70	05	1.80	0.0197
2	6	70	10	1.70	0.0186
3	6	70	15	1.40	0.0153
4	6	80	05	2.40	0.0263
5	6	80	10	1.95	0.0213
6	6	80	15	1.40	0.0153
7	6	90	05	2.00	0.0219
8	6	90	10	1.60	0.0175
9	6	90	15	1.10	0.0120
10	8	70	05	2.30	0.0252
11	8	70	10	2.42	0.0265
12	8	70	15	1.30	0.0142
13	8	80	05	2.20	0.0241
14	8	80	10	1.50	0.0164
15	8	80	15	1.58	0.0173
16	8	90	05	1.57	0.0172
17	8	90	10	1.20	0.0131
18	8	90	15	1.10	0.0120
19	9	70	05	2.20	0.0241
20	9	70	10	2.40	0.0263
21	9	70	15	3.20	0.0350
22	9	80	05	1.50	0.0164
23	9	80	10	2.20	0.0241
24	9	80	15	2.82	0.0309
25	9	90	05	1.1	0.0120
26	9	90	10	1.8	0.0197
27	9	90	15	1.6	0.0175

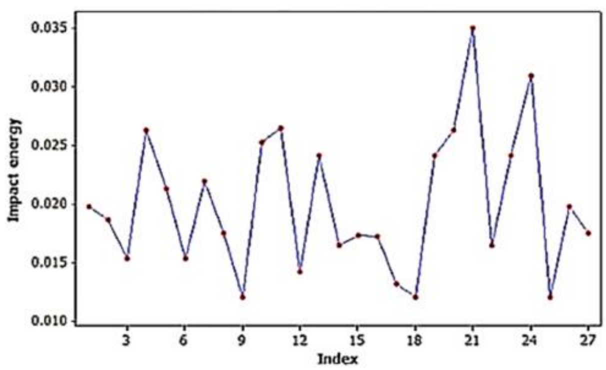

Fig. 9 Time series plot for impact energy

2. Time Series Plot for Impact Energy

Fig. 4 reveals the time series plot of impact energy for the all the specimens which are coated under electroless Ni-P with different parameters. From the graph, it can be observed that, at the index value of 21 and 24, the specimen coated with 15 % phosphate gives higher impact energy. It is also observed that the specimen coated with 5% phosphate gives average impact energy and index values of 3, 6, 9, 12, 17, 18, and 25 shows the lower values of impact energy.

3. Main Effects Plot for Impact Energy

Fig. 4 represents the influence of temperature, pH, and reducing agent on the electroless Ni-P coated substrate. It can be observed that impact energy decreases as temperature increases. It is deduced that, as pH increases, impact energy also increases.

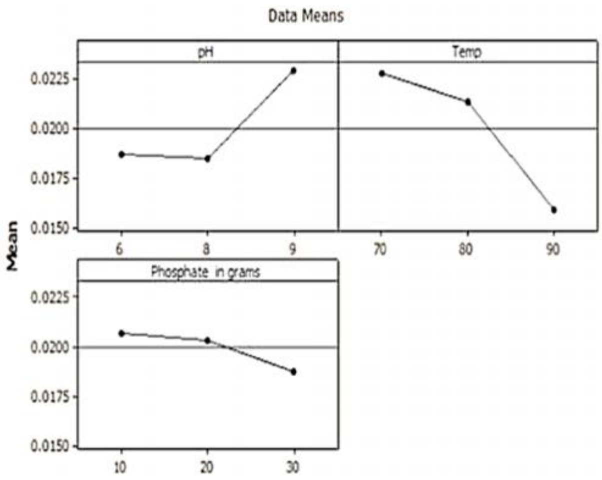

Fig. 10 Main effects plot for impact energy

4. Analysis of Variance (ANOVA)

The ANOVA results for electroless Ni-P coating are depicted in Table VIII. It is seen that concentration of pH has got the most significant influence on impact energy. The interaction (T*R) is found to have significant effect on the substrate characteristics of electroless Ni-P coating and interaction between bath temperature and concentration of (T * P) and concentration of phosphate (P×R) shows negligible percent of contribution in coating.

TABLE VIII
ANOVA RESULTS FOR IMPACT ENERGY

FACTOR	SS	Dof	Mean Sq	F	P %
pH	1.98	2	0.99	4.304348	15.63786
Temp	0.94	2	0.47	2.043478	4.938272
Phosphate (R)	0.15	2	0.08	0.347826	---
(T*P)	0.52	2	0.26	1.130435	0.617284
(T*R)	1.46	2	0.73	3.173913	10.28807
(P*R)	0.09	2	0.04	0.173913	---
Error	4.58		0.23		75.5144
Total error	9.72	20			100

Note: Dof-Degrees of freedom SS-sum of squares p-percentage of contribution

V. CONCLUSIONS

It is concluded that, the electroless Ni-P coating on medium carbon steel shows vital role on the performance of the coated component when subjected to the change in coating bath temperature, pH value, and percentage of phosphate. Electroless nickel coating done with parameters 9 pH, 70 °C temperature, and 15 percentage of phosphate shows an increase in the impact energy of the specimen. Because of poor adhesion at the interface, the specimen coated with higher temperature has yielded low impact energy. Based on ANOVA, pH contribution has shown maximum influence on the impact energy. Finally, it can be concluded that the bath parameters of the Ni-P coating have the influence on the strength of the specimen.

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