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Effect of Bath Parameters on Electroless Ni-P and Zn-P Deposition on 1045 Steel Substrate

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This research investigated the effect of varying bath concentration, operating temperature and plating time on the electroless deposition of Ni-P and Zn-P on the class AISI 1045 carbon steel. 0.4 g/L to 0.7 g/L for Ni^{2+} and Zn^{2+} concentration was used as bath formulation while other components were kept constant. Operating temperature was varied from 70 ^{0}C- 100 ^{0}C and the plating time was between 15 minutes and 35 minutes., It was observed that a bath concentration of 0.50 g/L -0.60 g/L for Ni-P, deposition time of 25-35 minutes and 80 ^{0}C temperature gave brightness and uniform film deposition on the carbon steel. Bath instability was observed as the temperature of the bath increased beyond 80 ^{0}C. Increased concentration of the metal source showed a down effect on the plating process and plating time below 25 minutes gave a dull bright nickel deposit. Zn-P maintains continuous bright deposition from 0.55 g/L to 0.70 g/L Zinc Chloride salt as the temperature increases from 70 ^{0}C to 100 ^{0}C. Increasing the deposition time gave a positive effect on the bright deposit of Zn-P on the substrate. It was also observed that increasing time of plating served to increase amount of deposit per unit area. Both coatings have good wear resistance at the best plating parameter. Scanning Electron Microscope result revealed continuous film surface morphology. The plating parameters obtained could be applied on low carbon steel for decorative or improved wear resistance where the cost of other method of coating is high-priced.

Keywords: Electroless, bath parameters, AISI 1045 steel, surface morphology

1. INTRODUCTION

Metal plating in the form of electroplating or electroless plating is the method of applying a metallic coating to another material [1]. There are many reasons to plate objects. These reasons include corrosion control, resistance to wear and decorative purposes [2]. Corrosion control is of essence in industries, as its destructive impact affects every aspect of human life [3-4]. Most engineering

materials for design include AISI 1045-steel which is of high tensile strength of magnitude 1.82 x 10^5 psi with carbon content between 0.43-0.50 percent C [5]. It is easily produced in the South West of Africa and possessed properties of interest for fabrication of medical appliances. This type of steel is a beneficent metal as its applications are vast. However, it is susceptible to corrosion and wears [5-6]. Protective coatings are probably the most widely used practice for corrosion control [7]. However, developing and studying electrolytes of deposit of Zn alloy is a high priority problem in electroplating [8]. The coatings barely give significant structural strength, but they protect other materials by preserving their strength, reliability and integrity. This can be achieved using different techniques. Electroless plating is a form of protective coating technique [9]. It is a chemical reduction reaction which occurs when metal ions are autocatalytic reduced in an aqueous solution containing a chemical reductant and the successive deposition of the metal without electrical current passed through it [10]. This can also be referred to as a redox reaction**.** Electroless nickel has shown its superiority for producing coatings with exceptional corrosion and wear resistance [11-12]. Coatings obtained from electroless plating technique are uniform and they have homogenous distribution despite of the substrate geometry. Electroless nickel coatings have excellent corrosion resistance in several industrial environments [13]. They are widely used either as protective or decorative coatings [14]. Electroless nickel does not perform as a sacrificial coating but behaves as a proper barrier coating, protecting the substrate by sealing it off from the corrosive environments [15-16]. The many benefits of electroless deposition are observed from its wide application in various fields [17]. Varying factors affect the deposition and appearance of the metal on the plated substrate of an electroless plating process. These factors include but not limited to: bath age, bath concentration, operating temperature, bath time, bath volume, and operating pH, bath Additives [17-18]. Bath concentration could be controlled by analysis of the depositing metal. A well-controlled electroless plating process gives better yield [19]. The deposition time of an electroless bath depends a lot on the stability of the bath. The more stable the bath is; the longer the plating duration and thus a continuous deposition of the metal as a result of replenishing the amount consumed per unit area [20]. Bath temperature is a very significant plating parameter in determining plating rate [21]. This depends on the material being plated. Operating pH is also a vital factor on deposited phosphorus content. In general, higher pH ranges indicates lower phosphorus content in the deposit, while lower pH values result in higher-phosphorus deposits [22]. Other bath elements that influence the deposition rate are complexing agents and stabilizers. Electroless plating is a process that is affected by various factors. The stability and equilibrium of the bath is essential. Nickel has shown outstanding success as to its application for electroless coating [23-24]. Zinc on the other hand has been applied more for other forms of coating, like electroplating, hot dip galvanization, galvanization and electrogalvanization. The benefits of Zinc and its vast availabilty sets it out for many enegineering applications [25]. The combine benefit of electroless plating and Zinc makes it essential to examine bath conditions for electroless zinc alloy depositions. A study of some of these factors that affect deposition rate and homogeneity of coating is also of increased importance. This study looks to deposit electroless nickel-phosphorus (Ni-P) and electroless Zinc-phosphorus (Zn-P) alloys on AISI 1045 steel for application of the fabrication of medical equipment, and observe the effects of some bath parameters; the concentration of the source of metals, the deposition time and temperature of the solution on the electroless Ni-P and Zn-P plating bath. Bath parameter conditions

for electroless Zn-P has not been out rightly stated, as electroless Zn-P has been given minimal consideration. This work studies these bath conditions in relation to electroless Ni-P bath with emphasis on the abrasion, amount deposited per unit area, film uniformity and film continuity and the physical appearance of the plated material.

2. EXPERIMENTAL PROCEDURE

2.1. Material preparations

Table 1. Chemical Composition of the as-received AISI 1045 Mild Steel

Elements:	Fe	Mn	C	Si	S	O
Wt%:	98.80	0.35	0.47	0.18	0.05	0.15

The mild AISI 1045 steel substrate of dimension 1 m × 20 mm, with thickness 1.5mm was used for this analysis. The chemical composition of the mild steel is as shown in Table 1. The mild steel substrate was sectioned into smaller pieces of dimension 15 mm × 10 mm and drilled with a 2 mm drilling bit so as to enable the sample to be held and immersed during the plating process. The samples were grinded and polished with emery paper to mirror like finish and rinsed with distilled water. Pickling of the samples were done in dilute H_2SO_4 acid solution to remove all organic contaminants and oxides. Then the pickled samples were washed in distilled water, dried and made ready for electroless plating.

2.2. Electroless deposition of Ni-P and Zn-P on AISI 1045 Steel

Table 2. Bath components for electroless nickel and zinc plating on 1045 steel substrate

Component	Function	Amount
Nickel Chloride	Source of Nickel metal	0.40 -0.70 (g/L)
Zinc Chloride	Source of Zinc metal	0.40-0.70 (g/L)
Sodium Hypophosphite	Reducing agent	0.30 (g/L)
Tri-sodium Citrate	Complexing agent	0.25 (g/L)
Ammonium Chloride	Buffer	0.50 (g/L)
Temperature		70^0C-90^0C
Deposition Time		15-35min
pH		4-5

The electroless plating was performed by immersing the mild steel substrate in a glass beaker containing 250 cm^3 of electroless plating solution which comprised of; source of Nickel metal (Nickel Chloride), reducing agent (Sodium Hypophosphite), a stabilizer (triSodium Citrate), a buffer (Ammonium Chloride). The reducing agent was used at 0.30 g/L, buffer and stabilizer at 0.50 g/L and 0.25 g/L respectively. The source of the metal salt was varied as shown in Table 2.
Bath setup include; thermometer, agitator, sample holder, bath solution, heat source, sample (AISI 1045 steel). The chemical reducing agent (sodium hypophosphite) in the solution provides the electrons needed to convert the metallic ions to the elemental forms.

The deposition of electroless Ni–P alloy was carried out using the formulated bath composition shown in Table 2. The sample was placed in a heating bath with the temperature controlled with a contact thermometer. The bath pH was adjusted with ammonium chloride acting as the buffer. The temperature of the autocatalytic reaction ranged from 70 ^{0}C to 90 ^{0}C, concentration 0.40 g/L-0.70 g/L of the metal source and the deposition time 15-35 minutes. The deposited weight was calculated by increase in the weight of the sample using the equation

$$M_i\,(g) - M_o\,(g) = M\,(g) \quad (1)$$

Where M_i is the weight of the sample after deposition, M_o is the initial weight of the substrate and *M* is the deposited weight.

2.3. Wear Resistance Test

Tribological wear analysis was executed via a CETR UMT-2 Tribometer with load of 25 N over a period of 1000 sec at a speed of 5 Hz. The wear analysis was a dry sliding wear test, using a pin on disc tribometer without the use of a lubricant. The coefficient of friction over time and the load exerted were obtained.

3. RESULTS AND DISCUSSION

3.1. Effect of temperature on the physical appearance of Ni-P and Zn-P plated sample

Table 3 present the effects of varied temperature and concentration of the bath on the physical characteristics of the Ni-P and Zn-P deposit on the AISI 1045 steel substrate. It has been proposed that at temperatures below 70^{0}C, electroless Ni-P depositions barely occur [26-28]. However, as seen from the open literature, specific bath conditions for electroless Zn-P deposition have not been determined; literature on this is rather scarce. Table 3 shows the weight of Ni-P and Zn-P deposited as temperature is increased from 70 ^{0}C to 100^{0}C. The physical appearances at higher temperatures of 85°C to 100°C were dark and black burnt for Ni-P deposits, but gave bright appearances for the Zn-P deposits. It reiterates the effect of increased temperature on the electroless nickel bath and indicates that electroless Zn-P plating can be conducted at higher temperatures than that of nickel.

Table 3. Physical appearance of deposited Ni-P and Zn-P on AISI 1045 steel for varied temperature and varied concentration of Ni^{2+} and Zn^{2+}

Temperature °C	Ni-P deposits	Zn-P deposits	Concentration g/L Ni^{2+}/ Zn^{2+}	Ni-P Deposit	Zn-P deposits
70	Dull bright	Dull bright	0.40	Dull bright	Dull bright
75	Bright	Bright	0.45	Fairly bright	bright
80	Very bright	Very bright	0.50	Very bright	bright
85	Black	Very Bright	0.55	Very bright	Very bright
90	Black	Bright	0.60	Very bright	Very bright
95	Black burnt	Bright	0.65	Bright	Very bright
100	Black burnt	Bright	0.70	Bright	Very bright

3.2. Effect of concentration and deposition time on the physical appearance and amount per unit area of electroless Ni-P and Zn-P plated sample

Table 4. Weight and Physical Appearance of deposited nickel on AISI 1045 mild steel for varied deposition time

Deposition Time (min)	Physical Appearance of Ni-P deposits	Physical Appearance of Zn-P deposits
15	Dull bright	Bright
20	Bright	Bright
25	Bright	Bright
30	Very Bright	Bright
35	Very Bright	Bright

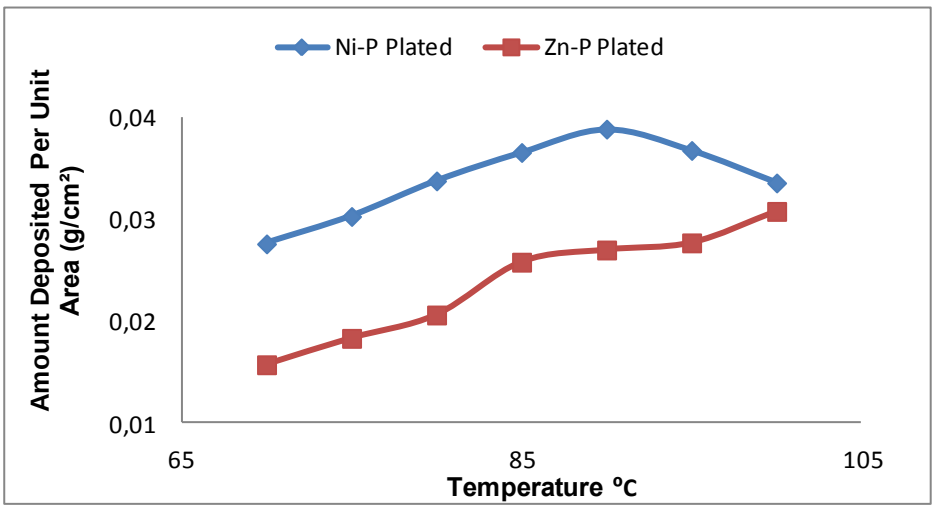

Figure 1. Amount deposited per unit area of Ni-P and Zn-P deposited on AISI 1045 steel at varied Temperature

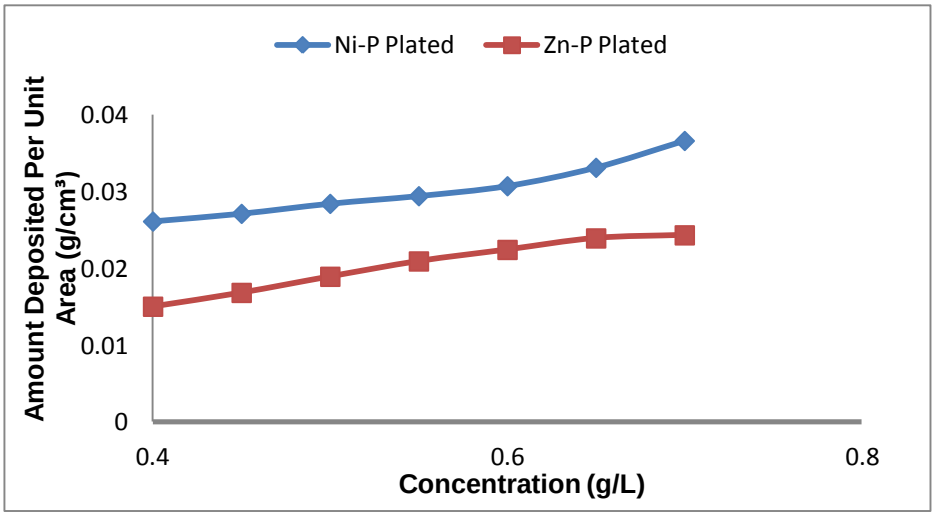

Figure 2. Amount deposited per unit area of Ni-P and Zn-P deposited on AISI 1045 steel at varied concentration

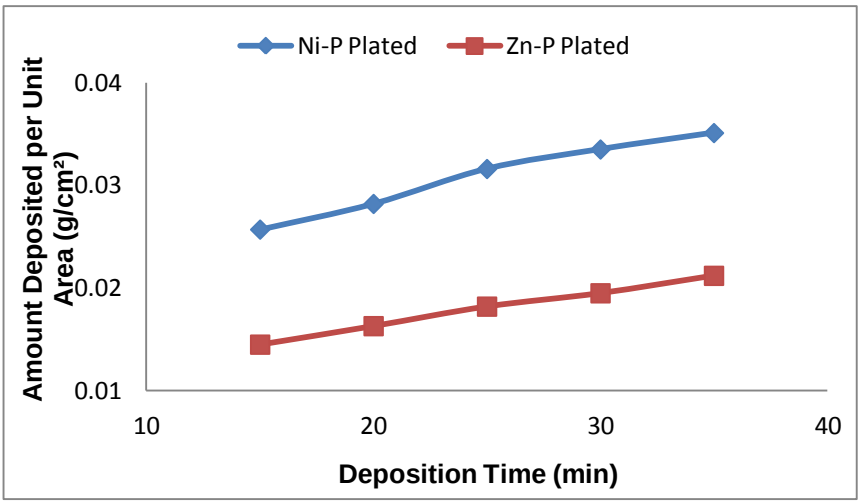

Figure 3. Amount deposited per unit area of Ni-P and Zn-P deposited on AISI 1045 steel at varied deposition time

The amount (grams) per litre of the source of Ni metal (Nickel Chloride) and Zn metal (Zinc chloride) was varied with temperature at 80 ^{0}C, deposition time at 30 minutes and other bath parameters maintained. The effect of the concentration on the plating appearance is shown on Table 3. Table 4 reveals the observed physical appearance of the substrate after Ni and Zn deposition at a deposition time range from 15 minutes to 35 minutes. Bright finishing was observed as other bath parameters were maintained. Figures 2 and 3 showed an increase in the deposited weight per unit area as concentration and deposition time increases. Hence a proportionate effect of amount deposited with time increase is observed. From the physical observation, as the deposition time exceeds 25 minutes, the appearance of the plated Ni-P and Zn-P samples remained bright. The plating continues as time is

increased, this can be attributed to the stability of the process. Electroless plating depends strongly on the stability of the bath. This is of importance in industries where plating is done for decorative purpose; a bright finishing is essential. It can be observed from Figure 3 that the amount per unit area deposition of the electroless nickel and zinc coating has been influenced by the immersion time. The general trend seen from the Figure indicates that with the increase of immersion time the coating weight keep on increasing through the successful increase in the plating time. Also observed is the quantity of Ni deposit is higher than Zn deposit at the same operating parameters. This might be due to force of attraction or bonding force between Ni and Fe being higher than bonding between Zn and Fe. Figure 7 showing SEM/EDS confirmed more of Ni deposit than Zn on the same substrate and operating parameters. Amount or thickness of deposition control could be easily selected with Ni and Zn electroless deposition on AISI 1045 steel substrate.

3.3. Effect of temperature on the amount deposited per unit area of Ni-P and Zn-P on substrates

Figure 1 presents the Amount deposited per unit area of Ni-P and Zn-P deposited on AISI 1045 steel at varied Temperature. The temperature range of the bath is observed between 70 ^{0}C – 100 ^{0}C. A continuous increase in the amount deposited per unit area was observed for both Ni-P and Zn-P with increase in temperature until 85 ^{0}C – 100 ^{0}C where a decline in the weight of Ni-P deposited was observed, while the weight of Zn-P deposited increases linearly with temperature. The effect of temperature on the electroless bath is very essential as it produces the energy needed for the chemical bonding occurring from the metal oxidation. This shows that the process takes place quickly as the heat generated increases by the increase in the temperature. However, higher temperature above 80 ^{0}C does not favoured electroless Ni-P deposition at 0.55 g/L Nickel chloride content indicating that higher concentration may be needed for the bath stability for an electroless plating to occur satisfactory.

3.4. Effect of electroless Ni-P and Zn-P on tribology of AISI 1045 steel

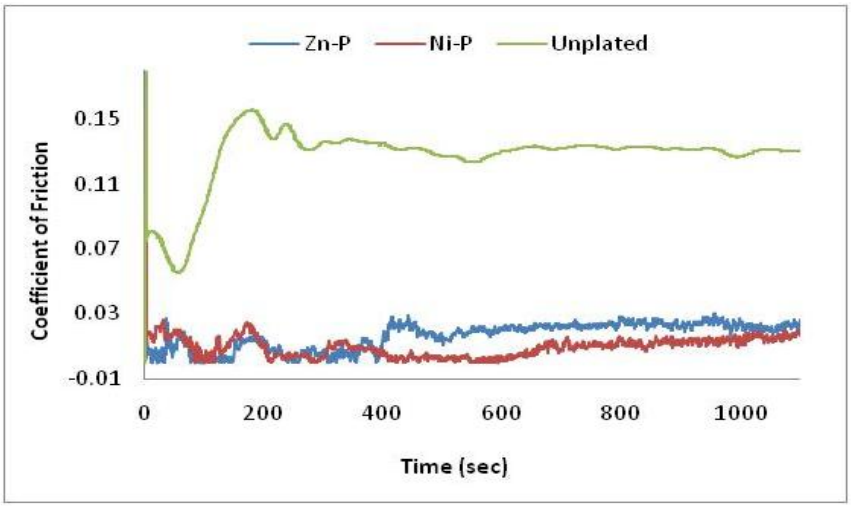

Figure 4. Plot of Coefficient of Friction Vs Time (sec) for electroless Ni-P and Zn-P deposition on AISI 1045 steel substrate

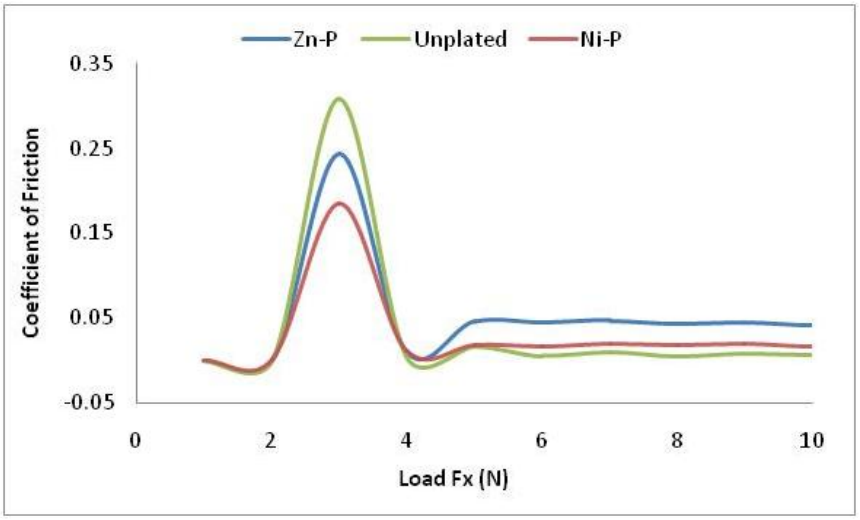

Figure 5. Plot of Coefficient of Friction Vs Load Fx (N) for electroless Ni-P and Zn-P deposition on AISI 1045 steel substrate

Figures 4 and 5 present the coefficient of friction with time of revolution and with load function respectively for the unplated and plated steel substrate. Coefficient of friction is more pronounced for the unplated substrate than Ni and Zn electroless plating as shown in Figure 4 and 5. The wear rate for both nickel and zinc electroless plating on AISI 1045 steel substrate is very low which indicates that with little lubrication it could serve well in design of engine parts rotating against each other. The coating consists of a nickel phosphorous alloy containing 12.76 percent phosphorous, some trace impurities, and the balance nickel while zinc phosphorus amount to 11.72 percent (Table 5). Good frictional properties are produced by the phosphorous content, which provides natural lubricity, helps minimize heat buildup, and reduces scoring and galling [29]. From Figure 5 the coefficient of friction of electroless nickel-plated steel and zinc plated steel is less than 0.05 and is about 0.2 when plotted with load function. It is projected that the good wear resistance found on Ni-P plate could be due to the work hardening effects, good adhesion and higher hardness value of Ni. However, low wear resistance (slight difference) found on the Zn-P could be due to: Lower hardness value of the Zn, Traces of micro-cracking and pullout observed in the coating microstructure. It is also observed, that the wear trend of both plated and the unplated sample changed from 4 to 10N. This behaviour could be due to the adverse effect of the load (i.e high peak observed at 2N-4N). This suggests that the load used has penetrated via the coating surface to the substrate at that region in agreement with the high pull-out and grooves observed in the SEM images of the worn surface in Figure 9. Form the results, an extensive damage, ridges shape were found on the unplated substrate. The worn coated Zn-P surface revealed extensive cracking, plastics deformation, pullout and potholes while Ni-P surface indicate mild abrasion occurring.

3.5. SEM/EDS analysis of electroless Ni-P and Zn-P deposited on AISI 1045 steel substrate

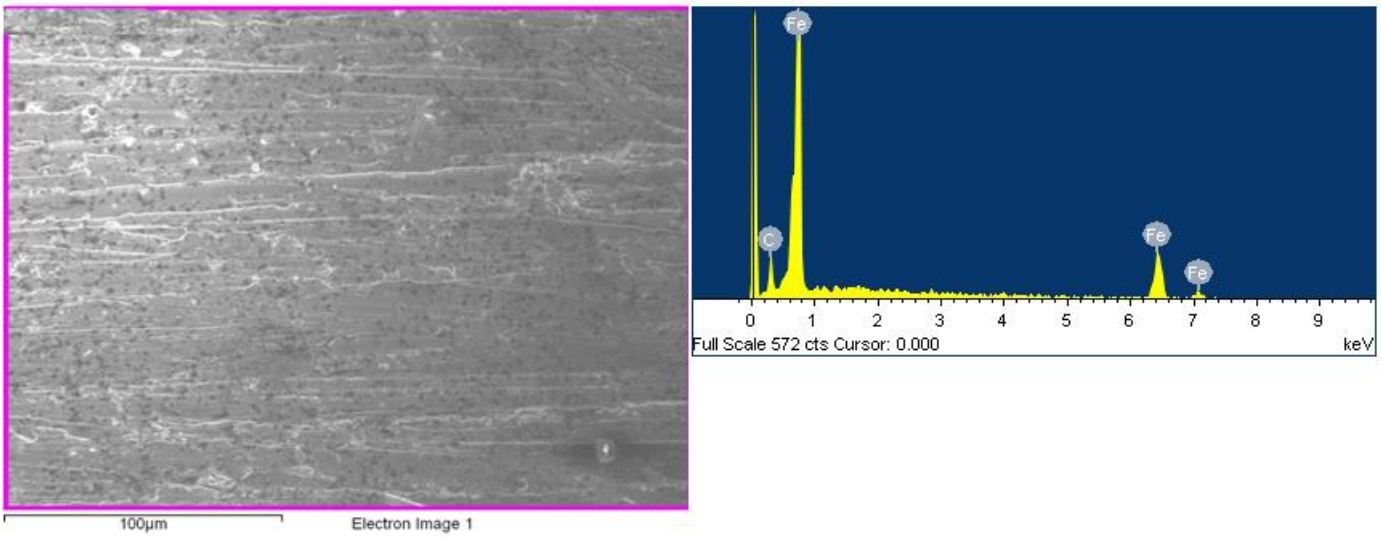

Figure 6. SEM Micrograph and EDS spectra for Unplated AISI 1045 Steel

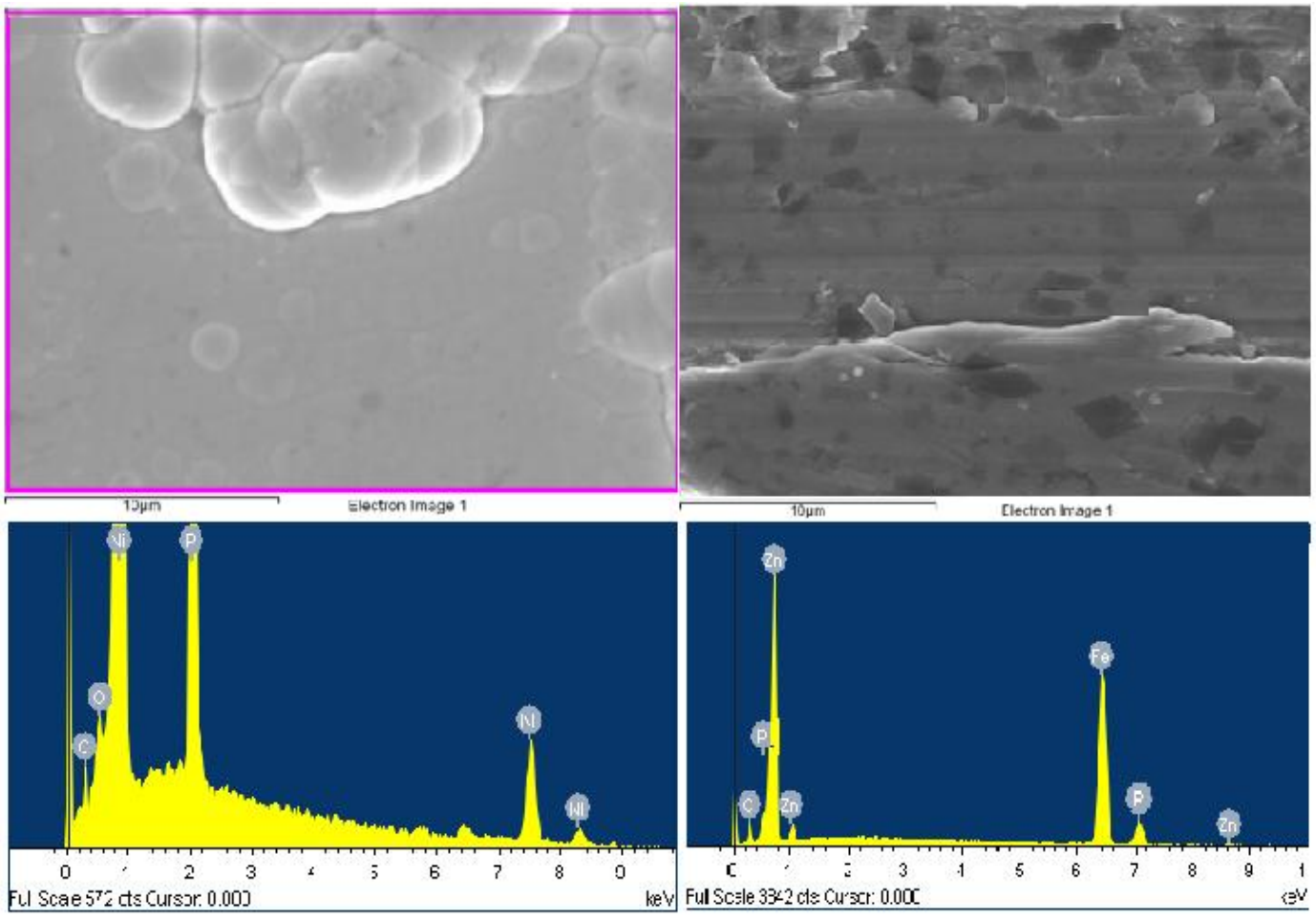

Figure 7. a) SEM for Ni-P b) SEM for Zn-P and EDS analysis c) for Ni-P EDS analysis at 0.55 g/L concentration, 80^{0}C Temperature and 25 minutes

SEM analysis of the samples was obtained using a field emission scanning electron microscope (Model JEOL JSM -7600F). Microprobe analysis was performed using energy dispersive spectrometry (EDS). The SEM micrograph and EDS spectra for the unplated mild steel substrate is shown in Figure 6. The surface of the substrate is plain and without cracks or defects. The plated samples were also observed for physical appearance.

Figure 7 shows the SEM/EDS of effect of temperature on the surface morphology of the electroless Ni-P and Zn-P plated mild steel at 80 ^{0}C and 25 minutes plating time. At 80^{0}C, the EDS reveal the film components in weight% of nickel and phosphorous deposited were 78.74% and 12.76% respectively and the weight of zinc and phosphorous was 75.61% and 11.72 %. Deposition of the alloys at this temperature gave considerable yield. Film overlaps was observed at both deposition (Fig. 7a and b) which may be due to lack of proper agitation of the solution although this enhances the wear resistance of the deposit.

3.6. Effect of concentration and deposition time on the surface morphology of Ni-P and Zn-P deposited on AISI 1045 steel substrate

Figure 8 shows the SEM/EDS of the electroless Ni-P and Zn-P plated mild steel at 0.55 g/L concentration and at 30 minutes deposition time. Figure 7a indicates uniform deposition with nodular arrangement of nickel film while Figure 7b reveals feather like film deposition of Zn on the substrate. The growths of spherical crystallites of nickel film and feather like crystal of zinc covered the entire surface, thereby giving it a uniform appearance, finer grained structure and dendrites free.

Table 5. Composition of the Ni-P and Zn-P film electroless plating on AISI 1045 steel substrate at 0.55 g/L concentration, 80 ^{0}C, 25 minutes and 30 minutes

Element	Weight% (Ni-P) 25 minutes	Weight% (Zn-P) 25 minutes	Weight% (Ni-P) 30 minutes	Weight% (Zn-P) 30 minutes
C	0.25	0.32	0.13	0.25
O	1.20	1.93	0.00	3.71
P	12.76	11.72	12.35	9.45
Fe	7.05	10.67	0.78	9.78
Ni	78.74	0.00	86.74	0.00
Zn	0.00	75.61	0.00	76.81

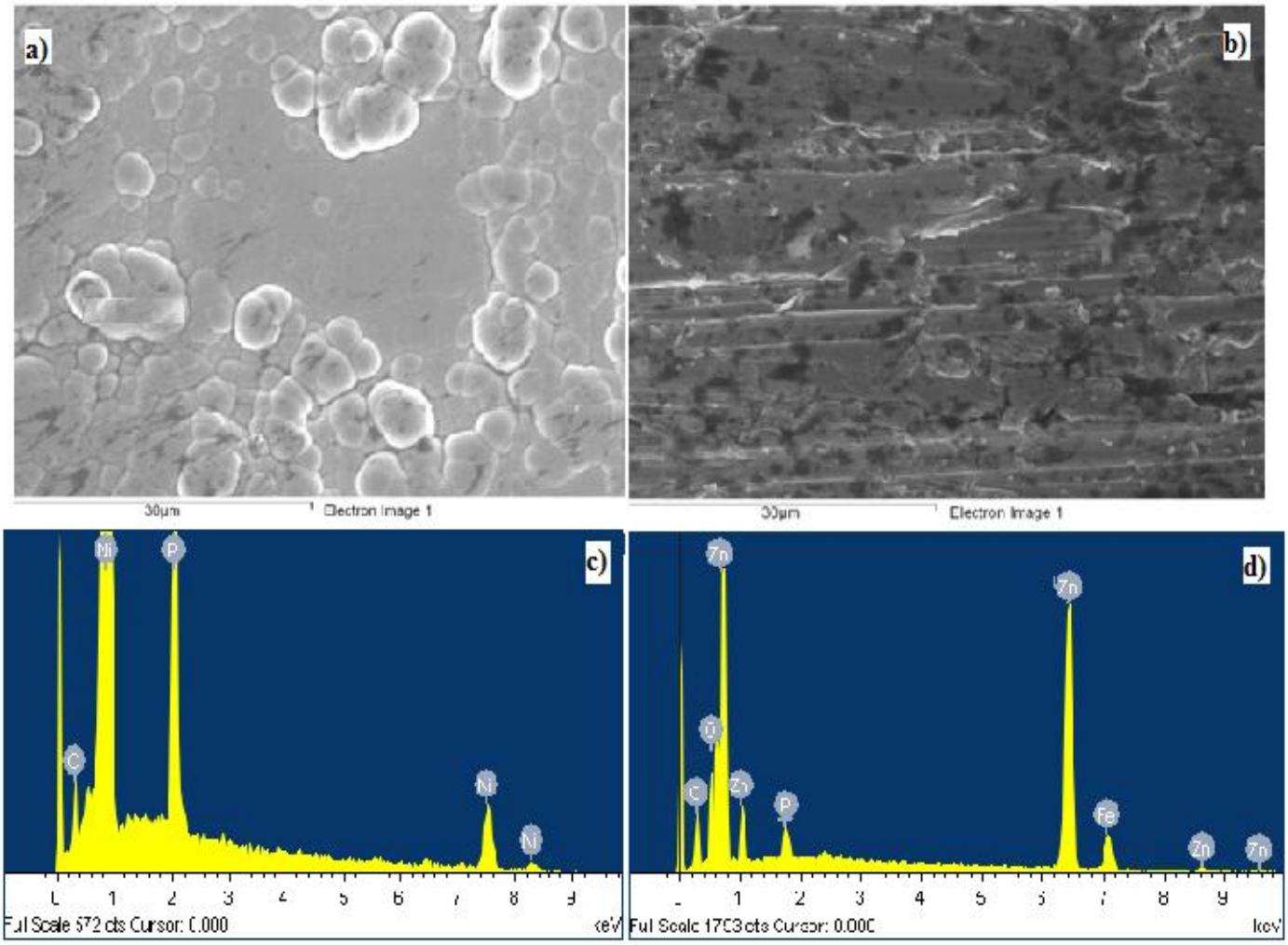

Figure 8. a) SEM for Ni-P b) SEM for Zn-P and EDS analysis c) for Ni-P EDS analysis for d) Zn-P at 0.55 g/L concentration, 80 ^{0}C temperature and 30 minutes deposition time

The EDS spectra showed high peaks of nickel and phosphorous elements, indicating the deposition of the alloy. This is further shown in Table 5 from the weight % of each element present. 78.74% and 75.61% weight of Ni and Zn were deposited respectively at 25 minutes deposition time.

Figures 8 c and d shows the EDS of the film deposited on the surface of the substrate for more quantitative analysis of the deposit interface and indicating the topography level of the Ni-P and Zn-P respectively. Table 5 shows the weight % obtained from the EDS analysis, indicating the proportion of the Ni-P and Zn-P deposited. Observed from Table 5 at 30 minutes plating time, Ni had an increase of 8% weight deposit above what was recorded at 25 minutes while Zn had 1.2 % an indication that time has more positive effect on the amount of Ni deposited than Zn on AISI 1045 steel substrate. This however, formed an advantage for using nickel to build up worn out parts in machineries.

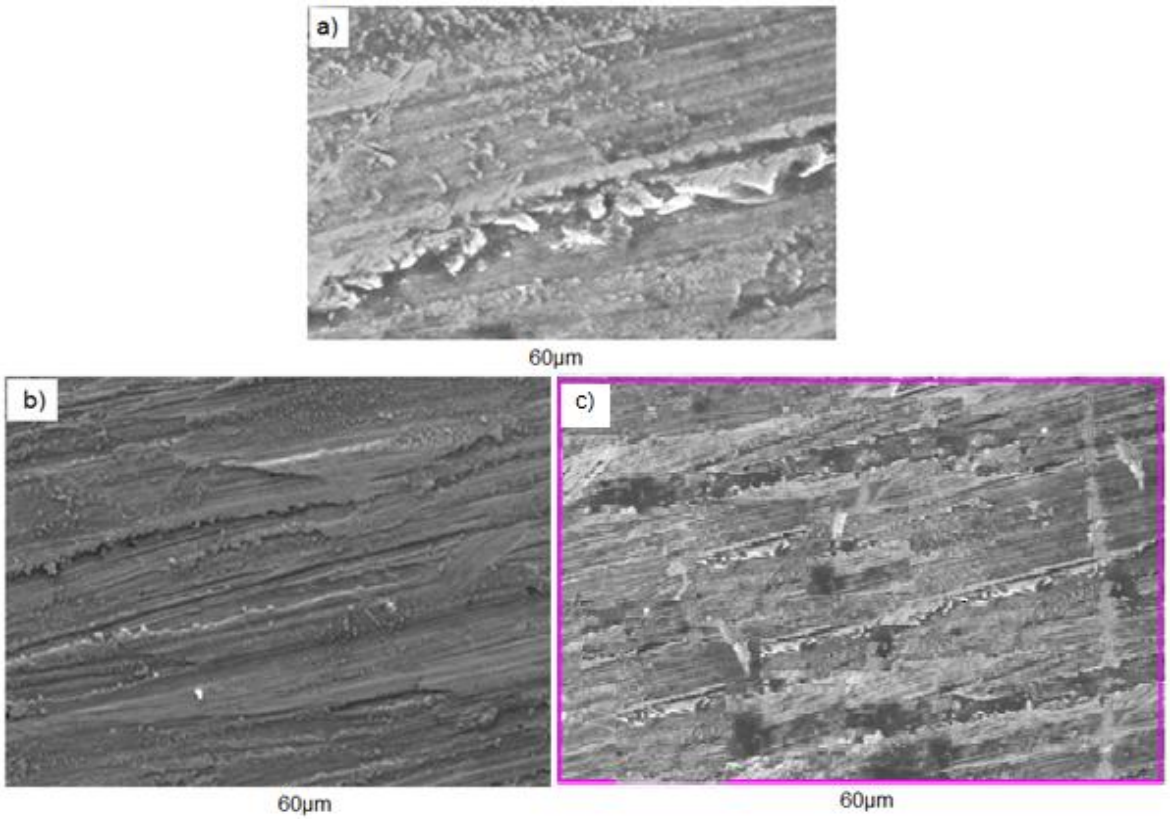

Figure 9. SEM of Worn Surfaces for a) Unplated b) Ni-P plated and Zn Plated AISI 1045 Mild Steel

4. CONCLUSION

From the study of the effect of concentration, temperature and deposition time on the electroless plating of Ni and Zn on low carbon steel type AISI 1045 steel substrate the following conclusions were made.

1. Increased concentration of the nickel metal source in the electroless plating process without a commensurate increase in other bath parameters will shift the equilibrium of the bath. This could also lead to breakdown of the metal before the deposition process is completed
2. Deposition time has a great influence on the plating process. It is observed that as long as the stability of the bath is sustained, amount deposited increases with increase in the plating time.
3. Temperature increase above 80 ^{0}C affects the bath significantly, the weight of the Ni-P metal deposited is seen to decline as reaction 'plate out' occurs. Also the physical appearance of the plated surface is dark suggesting the bath was unstable.
4. Optimum conditions for both Ni-P and Zn-P deposits were observed at concentration of 0.50 g/L - 0.60 g/L, deposition time 25-35 minutes and temperature70 ^{0}C-80 ^{0}C. However, temperature values for Zn-P deposits ranged higher from 70 ^{o}C – 100 ^{o}C as the appearance of the deposit looked bright. It was observed that the weight of nickel and zinc alloy deposited at these conditions was consistent and the physical appearance was bright and very bright.

5. The optimum parameters obtained for depositing electroless Ni-P and Zn-P on AISI 1045 steel could be 0.50 g/L - 0.60 g/L, deposition time 25-35mins and temperature 70 ^{0}C-80 ^{0}C for fabrication of medical equipment.
6. The SEM images showed the Ni-P deposits, gave nodular rings and while Zn-P deposits shows layers with uniform film, continuity, no cracks or defects.
7. The Ni-P and Zn-P film deposit enhanced the wear resistance of the AISI 1045 steel substrates therefore; it could be used for the designing of parts desiring more lubrication. The parameters obtained in this study could be employed for surface treatment of AISI 1045 steel where other methods of metal coating are high-priced.

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