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Optimization of the Concentration Bath of Ni-P Electroless Plating by Photon Energy

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Abstract: Electroless nickel (EN) plating is a chemical reaction process which depends upon the catalytic reduction process of nickel ions in an aqueous solution (containing a chemical reducing agent) and the subsequent deposition of nickel metal without the use of electrical energy. This method distinguished by good mechanical properties also high resistance to corrosion also very stable and uniform thickness deposits on over sides of surface sample. This work involves study the optimization the concentration bath of the Ni-P electroless plating by exploiting the photonic energy (UV radiation). As an assistant source of energy to decrease the temperature of bath water of coating and compared to heating only. The present work investigates the influence of bath compositions, which included Nickel sulphate hexahydrate, Sodium hypophosphite monohydrate, Thiourea, Lactic acid, Propionic acid, on the process of electroless Ni–P coating. The deposition rate as well as the bath stability were monitored to optimize the plating bath conditions with the different composition. The thickness of deposit layer was obtained by using a sensitive balance and permascope apparatus. The resulted Ni-P layer was uniform and stable and good mechanical properties.

Keywords: Ni-P electroless plating, reducing agent, concentration, UV radiation.

I. INTRODUCTION

The discovery that some alloys produced by electroless deposition, notably nickel phosphorus, have unique properties. The most popular metal used in the electroless plating being Nickel, so it is chosen for further investigation in this study.

Modern electroless plating began in 1944 with the rediscovery that hypophosphite could bring about nickel deposition. Subsequent work led to the first patents on commercially usable electroless nickel solution. Although these solutions were very useful for coating metals, they could not be used on most plastics because the operating temperature was 90-100 °C(1). The electroless plating process is achieved to improve the mechanical properties of the metal. Hiratsuka et al. have studied the effect of the Ni-P electroless plating on the wear properties of the metals (2).

The electroless Ni-P alloy deposit has been extensively used in electronics, machinery, automobile, aerospace, oil and gas production, and valve industries for its excellent properties, such as hardness, corrosion resistance, and wear resistance. The deposition rate of electroless nickel plating with hypophosphite as a reducing agent is generally below 20 µm/hr, it is thus important to search for effective accelerators in EN plating. It was observed that thiourea and other sulfur containing compounds can increase the rate of nickel deposition when present in very small concentrations(3).

The effects of two electroless nickel bath stabilizers; thiourea and maleic acid were investigated. The EN deposits were characterized in terms of surface morphology, microstructure, phase, and composition. It was demonstrated that the addition of thiourea and maleic acid significantly improved the stability of the EN bath. It was observed that the stabilizers have a minor and a major effect on phosphorus content and morphology of the EN deposits, respectively (4).

The effect of thiourea and lead acetate on deposition rate crystallinity, surface morphology, and phosphorus content of electroless nickel deposit. In the investigated pH range (4-5), thiourea gave rise to higher deposition rates yet lower phosphorus contents than lead acetate. The results of Scanning electron microscope (SEM) and Atomic force microscope (AFM) investigation showed that coarse nodule and relatively rough surface morphology were obtained in the presence of thiourea. A higher pH value as well as a greater thiourea concentration tends to enhance the crystallization of the electroless nickel deposit(5).

The effect of UV irradiation on nickel plating during the electroless plating process was investigated. Both enhancement of the plating rate and the quite opposite effect, suppression, were obtained. Either enhancement or suppression can be caused by selecting the step at which the UV light is irradiated on the substrate. By using these effects, a pattern can be made on nickel film without the etching technique. This technique is similar to the so-called etchingless technique used to make semiconductors (6).

Electroless Ni-P coating is widely used in industrial engineering applications due to its ability to alter and improve the surface properties of the steel substrates. Electroless nickel coating introduce an excellent combination of surface properties, where, it could add brightness, luster, and good appeal. The final coating layer also possess a very good adhesion with the coated substrates, this is the reasons for using such layer as an 'undercoat' for other coatings. The ability to produce a very homogenous composition and produce coating with a high corrosion resistance are mainly based on the plating bath composition. The present work investigates the influence of bath compositions, which included nickel sulphate, sodium hypophosphite and tri-sodium citrate, on the process of electroless Ni–P coating. The deposition rate as well as the bath stability were monitored to optimize the plating bath conditions with the different composition. The results of this work showed that the deposition rate of coating layer increase with the increase of nickel source and reducing agent while; sodium citrate concentration in the plating bath has adverse effect on the deposition rate of coating (7).

II. MATERIAL AND METHODS

2.1. MATERIALS

The chemicals were used in experimental work of electroless plating bath shown in the Table(1)

Table 1. Material for the composition bath (8)

Bath composition	Formal	Concentration (g/l).
Nickel sulphate hexahydrate	NiSO4.6H2O	21.2
Sodium hypophosphite monohydrate	NaH2PO2.H2O	24
Lactic acid	C3H6O3	28
Propionic acid	C3H6O2	2.2
Thiourea	CH4N2S	0.0008

Nickel sulphate hexahydrate was used as the source of nickel. Sodium hypophosphite was used as the reducing agent, which also serves as the source of phosphorus in the coating. Lactic acid and propionic acid were used as the complexing agents to control the rate of release of free metal ions for the reduction reaction. Thiourea was used as additives in the plating bath. Among them, thiourea was used as stabilizers to prevent the decomposition of the plating bath.

2.2 Sample Preparation

The used iron specimen was produced by LIBYAN IRON & STEEL CO., Misurata, with special specification. The chemical compositions of steel metallic piece were included on Fe, C, Si, Mn, P, S and Al with 99.402, 0.095, 0.22, 0.207, 0.014, 0.001 and 0.061 respectively. The assay (sample) is prepared (40mm $\times$ 30mm $\times$ 1mm) and pierced from above to be hanged inside the cup so that precipitation happened all over the sides. Diluted nitric acid is used to clean the surface and as a catalyst for the precipitation process. Deionized water is used afterwards and lastly with acetone many times and dried well and then be weighed.

2.3 Measurements and characterization

In this work, in order to investigate, how change in various parameter concentration bath when using photonic energy for electroless plating alloy from Ni-P, they will affect the deposition. The samples obtained were characterized by SEM, techniques. The operation parameters of electroless Ni-P deposition bath are shown in Table (2).

Table 2. Operating Conditions

PH	6.5
Temperature	80°C
Duration	60 min

The instrument as UV exposure box (Radio Spares, model No.556-238) used to exposure the UV light on the baths. Permascope (D211D type D211D NO.03112864), Scanning electron microscope, sensitive level balance were used to investigate the composition of deposition layers.

III. Results and Discussion

3.1 Thickness coat Measurement

Measurement the thickness of deposited layer by two methods:

Using a sensitive balance and using permascope apparatus. The values of thicknesses of deposition layer by using sensitive balance and permascope apparatus is very nearly. The deposit layer has good mechanical properties.

Thickness coating can be measurement by the following:

Using the sensitive balance:

After drying the sample so well, then weighed and the thickness of the coat is determined according to the following equation:

Thickness (cm)=(Weight of deposition (g)/(Density(g/cm^3) $\times$ Area(cm^2)) (1)

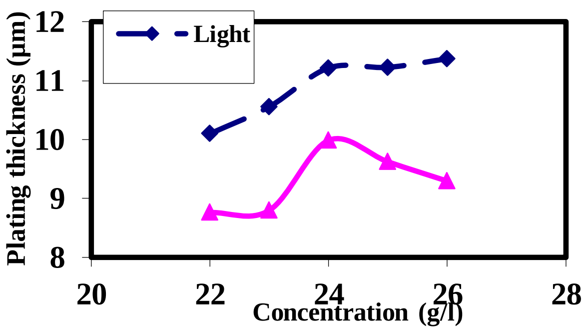

Figure1.Effect of change concentration Sodium hypophosphite monohydrate ($NaH_2PO_2.H_2O$)on plating thickness at (T=80˚C,t=60min,pH=6.5).

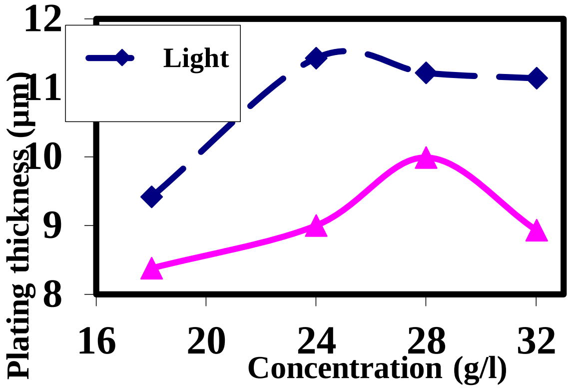

Figure 2.Effect of change concentration lactic acid ($C_3H_6O_3$) on plating thickness at (T=80˚C,t=60min, pH=6.5).

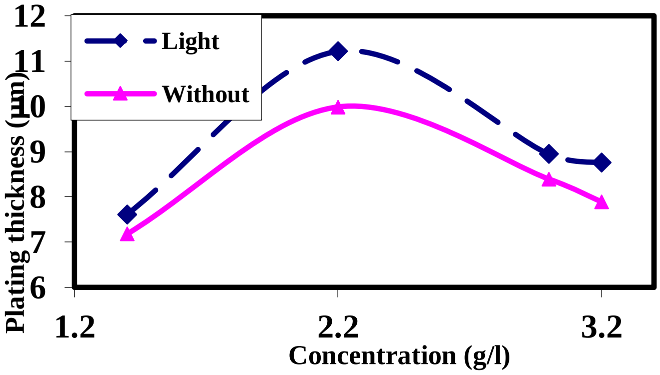

Figure 3.Effect of change concentration propionic acid ($C_3H_6O_2$) on plating thickness at (T=80˚C,t=60min, pH=6.5).

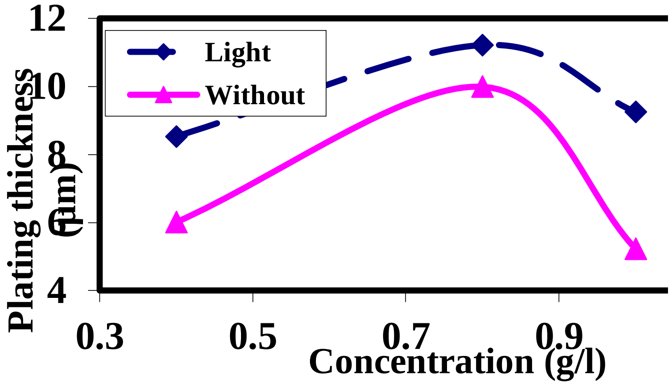

Figure 4. Effect of change concentration Thiourea (CH_4N_2S) on plating thickness at (T=80˚C,t=60min, pH=6.5).

Concentration of Nickel sulphate hexahydrate. In the presence light of maximum concentration is 22g/l and pH (6.5) or maximum concentration at without light21.2g/l .The effect of light on plating thickness is greater in the presence of light. The reason due to effect change concentration on reaction mechanism or also effect light on deposition.(ALjaray faiza,(12)). the change of concentration Sodium hypophosphite monohydrate ($NaH_2PO_2.H_2O$) from 22g/l to 26 g/l at pH (6.5) note increasing in plating thickness at present light This might be justified as increasing the concentration of hypophosphate the plating of phosphorous increases.

The thickness and concentration of Lactic acid ($C_3H_6O_3$) from 18 g/l to 32 g/l a, that addition lactic acid accelerates the rate of deposition up to 24 g/l and started to lower the rate of deposition at concentration 28 g/l. and this is because of the reaction mechanism in solution. And optimum plating thickness at present light with concentration (24 g/l) and at without light with concentration (28g/l) is thickness in presence light high from without light. De Minjer and Brenner,(9) studied the relationship between concentration of different acid especially lactic acid with the plating rate, is obtained high rate deposition at concentration 27 g/l at without light.

The plating thickness with concentration of propionic acid ($C_3H_6O_2$) from 1.4 g/l to 3.2 g/l the optimum concentration increased from plating thickness 2.2 g/l in present light and without light. At that addition propionic acid accelerates the rate of deposition up to 2.2 g/l and started to lower the rate of deposition at concentration 3 g/l. and this is because of the reaction mechanism in solution.

Lactic acid and propionic acid were used as the complexing agents to control the rate of release of free metal ions for reduction reaction Baskaran et al,(8).

The addition of thiourea in the concentration range (0.4 – 0.8 ppm) increases in presence or absence of light. The plating thickness started to decrease at concentration 1 ppm. The maximum thickness was noticed at concentration 0.8 ppm in presences or absence of light. From results Baskaran et al,(8). The addition of thiourea accelerates the rate of deposition up to 0.8 ppm and started to inhibit at a concentration of 1ppm. Thiourea can lower the activation energy of the electroless nickel deposition reaction.

The thiourea exerts an accelerating or inhibiting effect on the plating rate, depending on the thiourea concentration Talmey and Gutzeit, (11) and De minjer and Brenner,(10).on the sample.

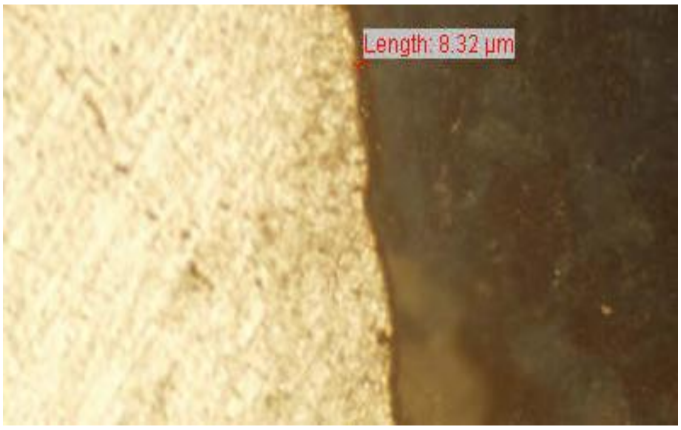

Figure5.SEM of Cross section view of an electroless plating

IV. Conclusion

The results obtained in this work for study the effect of exploiting the photonic energy on the Ni-P electroless plating. Also study the change concentration bath affected on deposition and measurement the thickness of deposited layer by two methods using a sensitive balance and permascope apparatus. These readings are very nearly and indicate that thickness of plating is uniform at sample surface, also the plating is homogenous. The best of thickness deposition (plating Ni-P) at concentration bath by using light.

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