

# Experimental Investigations on Electroless Plating of Nickel on Steel

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**Abstract**— Electroless nickel plating on steel is carried out by a chemical reaction and without the use of an external source of electricity. In electroless nickel plating, nickel chloride will come as a metallic salt and is reduced by using Sodium hypophosphite as the reducing agent to nickel metal, which then is deposited on the steel. This process has now become an established industrial technique and gained increasing importance. By such means not only pure metals but also alloys can be deposited on ferrous and non-ferrous materials to improve their surface characteristics. By using appropriate pre-plate treatment methods, a wide range of plastics and ceramics can also be plated by electroless methods. These processes have particular advantages in the field where galvanic processes can only be used at great expense or where they fail completely. The present paper studies the effect of different process parameters during electroless plating of nickel on steel. The effect of bath temperature, immersion time and bath pH on plating thickness, and deposition rate has been recorded and presented in graphical form.

**Key words:** Electroless plating, Nickel, Electroless Bath, Deposition rate

## I. INTRODUCTION

Electroless plating is an auto-catalytic process where the substrate develops a potential when it is dipped in electroless solution called bath which contains a source metal of metallic ions, reducing agent, stabilizer and others. Due to the developed potential, both positive and negative ions are attracted towards the substrate surface and release their energy through charge transfer process.

Each process parameter has its specific role on the process and influences the process response variables. Temperature initiates the reaction mechanism which controls the ionization process in the solution and charge transfer process from source to substrate. In addition to this, the substrate is activated before dipping into the electroless bath and sensitized to initiate the charge transfer process. Actually in electroless metal deposition process, no external current supply is required to deposit material on a substrate.

Electroless deposition has the advantages of simplicity and feasibility over other processes. The amorphous boron is introduced to improve the adherence between coating and the substrate besides improving properties, like wear resistance, hardness, corrosion resistance and surface roughness.

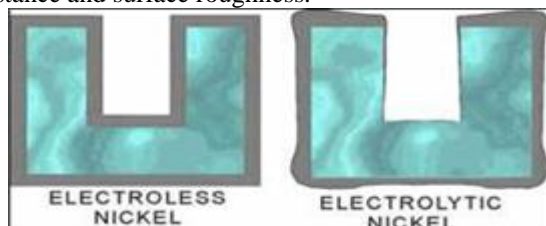


Fig. 1: Comparison between electroless and electroplating surfaces

The applications of electroless nickel have been reported in many industries, like petroleum, chemical, plastics, optics, aerospace, nuclear, electronic devices, computer, and printing because of its excellent corrosion and wear resistance properties.

The true electroless or “chemical” metal deposition processes are those capable of being used in place of Electro deposited coatings in that they permit a continuous metal deposition. Such deposits form only on certain catalytically active surfaces (auto catalytic deposition). The electrons needed to reduce the metal ions are provided by the reducing agents  $R^{n+}$  which surrender  $z$  electrons, themselves being oxidized to  $R^{(n+z)}$ . The equations for this are.

## II. LITERATURE REVIEW

**Mordechay Schlesinger and Milan Paunovic[1]**

Electroless (autocatalytic) plating involves the presence of a chemical reducing agent in solution to reduce metallic ions to the metal state. The name electroless is somewhat misleading, however. There are no external electrodes present, but there is electric current (charge transfer) involved. Instead of an anode, the metal is supplied by the metal salt; replenishment is achieved by adding either salt or an external loop with an anode of the corresponding metal that has higher efficiency than the cathode. There is therefore, instead of a cathode to reduce the metal, a substrate serving as the cathode, while the electrons are provided by a reducing agent. The process takes place only on catalytic surfaces rather than throughout the solution (if the process is not properly controlled, the reduction can take place throughout the solution, possibly on particles of dust or of catalytic metals, with undesirable results).

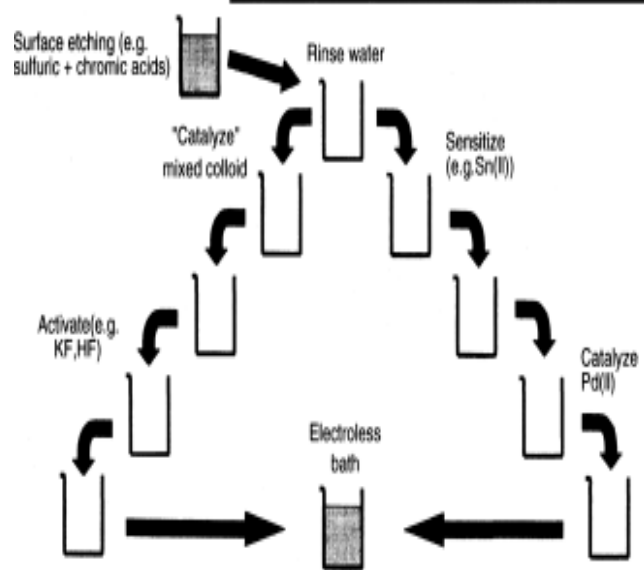


Fig. 2: Schematic representation of the electroless deposition process.

**Mallory and Hajdu[2]** studied the effect of bath pH and recorded this type results shown in figure.

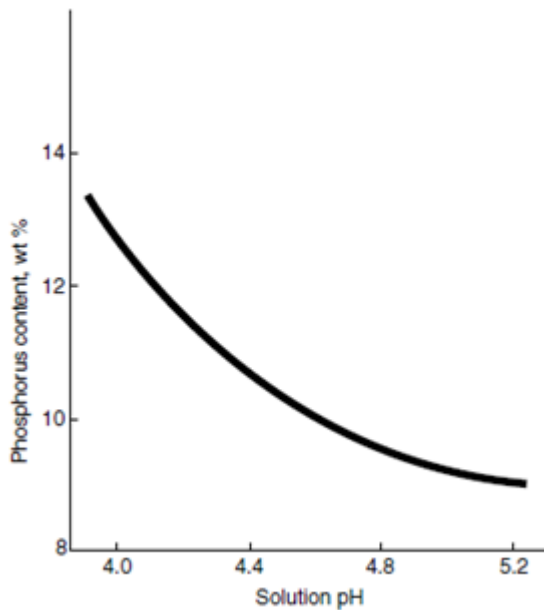


Fig. 3: Effect of pH on phosphorous content.

**I.E.Ayoub[3]** has studied on Electroless Ni-P Plating on Stainless Steel and concluded the following points.<sup>[1]</sup>

- (1) Ni-P coating can be successfully plated on the surface of stainless steel. The coating was compact, uniform and showed mixture microstructure of amorphous and microcrystalline with phosphorus contents of 8.57 wt.%.
- (2) The corrosion resistance of Ni-P on stainless is greatly improved by heat-treatment which were tested by the immersion experiment and the potentiodynamic polarization experiment in 3 % NaCl solution.
- (3) The micro hardness of the coating was increasing with increased the heat treatment temperature for a constant time (1hr), maximum hardness was achieved at 400°C.

**Juan Hajdu[4]** has developed Surface Preparation For Electroless Nickel Plating in which the masking is used in surface preparation.

#### A. Masking:

Many parts may require only partial coverage with electroless nickel, and portions of the substrate must be protected by masking. Many stop-off materials (tapes and coatings) used for electroplating can be applied for masking parts for electroless plating. Since most electroless nickel solutions operate at high temperatures, and the parts may stay in the bath for a long time, the masking materials should be tested thoroughly before using them on expensive parts.

All electroless plating solutions will perform best above minimum loading levels, which may be difficult to reach with masked parts.

Electroless coatings can be divided into three main categories like (i) alloy coatings, (ii) composite coatings and (iii) metallic coatings.[5]

Electroless coating technology is credited mainly to Brenner & Riddell. By the controlled chemical reduction reaction, the electroless coating chemistry has emerged as one of the leading growth areas in surface engineering, metal finishing etc. and is estimated to grow at a rate of

beyond fifteen percent per annum, certainly no other chemistry is growing at this rate.

Electroless coatings has unique physicochemical and mechanical properties for which they are being used increasingly

**Ashish Kumar Singh, Vishnu Kant Bajpai and Chetan Singh Solanki [6]** carried out electroless plating on c-Si solar cell.

In c-Si solar cell front contact metallization, nickel-copper electroplating scheme is found to be economical compared to other available techniques. In this metallization process, nickel seed layer deposition is looking simple but very important step in terms of its grain size, minimum thickness reaching the continuity of the film and its uniformity. Thin, uniform and continuous nickel seed layer helps in reducing the metal-semiconductor contact resistance as well as prevent junction shunting during silicide formation at the metal-semiconductor interface. Although, there are different process parameters affecting the morphology of the nickel film in electroless chemical bath deposition, but due to the photovoltaic effect of the p-n junction of the solar cell, the ambient light affects the nickel deposition process. The effect of light on electroless nickel (EN) deposition has been studied in this work. For this purpose the experiments have been performed in different lighting conditions like: dark, ambient, varying intensity UV-VIS light. Also, the nickel depositions have been done for different periods of time from very small period like 30s to longer periods like: 1 min, 2 min, 3 min and 4 min to see the effect at the initial stage of depositions as well as for prolonged deposition.

**J.C. Rajaguru, C. Au, M. Duke[7]** presented an investigation of electroless nickel plating on PerFactory™ rapid prototype model built on PerFactory™ R05 material. PerFactory™ R05 is acrylic based photo sensitive resin. It is a popular material in rapid prototyping using PerFactory™ method which employs additive manufacturing technique to build prototypes for visual inspection, assembly etc. Metallization of such a prototype can extend the application envelop of the rapid prototyping technique as they can be used in many functional applications. Unlike the electroless nickel plating on metal substrate, the process on acrylic resin substrate is not auto-catalytic. Hence, etching and activation are necessary for initiating the process. The final coating is then investigated using scanning electron microscope (SEM) together with energy dispersive spectroscopy (EDS) and x-ray diffraction (XRD) analysis to identify the morphology and structure of the coating. The SEM & EDS analysis on surface and chemical composition of model surface after each preliminary surface treatment are also presented. Finally the layer is tested on Vickers micro hardness tester.



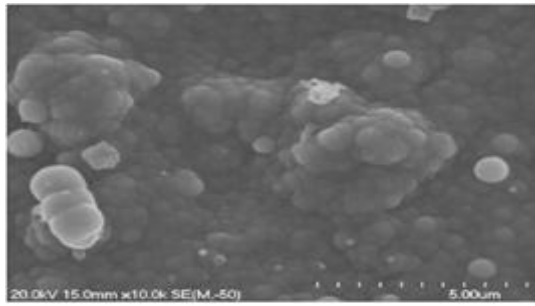


Fig. 4: The surface morphologies of original PerFactoryTM model surface, and surface after electroless nickel plating

Surface hardness of the nickel coating is measured using Vickers micro hardness tester, Leco LM700. Data are collected for the load range from 10 gf to 1000 gf for both nickel coating and the uncoated PerFactoryTM model using Vickers diamond indenter at 10 second dwelling time. Data are tabulated after getting the average of five readings for each loading condition in both cases. Fig. shows the Vickers hardness comparison. According to the hardness data, the nickel coated surface has higher hardness value than the uncoated surface for the loading range from 1000 gf to 200 gf. The highest recorded Vickers hardness is 14.7 HV at 500 gf loading which is 83% higher than without coating hardness value of 8 HV500 gf.

**Olawale Olarewaju Ajibola, Daniel T. Olorunfoba, and Benjamin O. Adewuyil [8]** examined effects of hard surface polishing grits and activation on electroless-nickel (EN) plating on cast aluminium alloy substrates in sodium hypophosphite baths. As-received aluminium alloy sample sourced from automobile hydraulic brake master cylinder piston was melted in electric furnace and sand cast into rod. The cast samples were polished using different grits (60  $\mu\text{m}$ – 1200  $\mu\text{m}$ ) before plating.

The effects on adhesion, appearance, and quantity of EN deposits on substrates were studied. Observation shows that the quantity of EN deposit is partly dependent on the alloy type and roughness of the surface of the substrates, whereas the adhesion and brightness are not solely controlled by the degree of surface polishing. The best yield in terms of adhesion and appearance was obtained from the activation in zincate and palladium chloride solutions. Higher plating rates ( $\text{g}/\text{mm}^2/\text{min}$ ) of  $3.01E - 05$ ,  $2.41E - 05$ , and  $2.90E - 05$  were obtained from chromate, zincate, and chloride than  $8.49E - 06$ ,  $8.86E - 06$ , and  $1.69E - 05$  as obtained from HCl etched, NaOH, and H<sub>2</sub>O activated surfaces, respectively.

**Prasanna Gadhari, and Prasanta Sahoo[9]** investigate the influence of coating process parameters on the microhardness of electroless Ni–P–Al<sub>2</sub>O<sub>3</sub> composite coating with the help of Taguchi analysis. Four parameters, namely, concentration of nickel sulphate as a nickel source, concentration of sodium hypophosphite as a reducing agent, concentration of Al<sub>2</sub>O<sub>3</sub> particles as concentration of second phase particles, and annealing temperature, are considered and fitted into an L<sub>27</sub> orthogonal array to find out the optimized condition for improved hardness of the coating. The optimized condition is found to yield about 20.47% improvement in hardness of the coating compared to the initial condition. The significance of the process parameters and their interactions on the hardness of electroless Ni–P–Al<sub>2</sub>O<sub>3</sub> composite coating is studied with the help of analysis

of variance, which revealed that annealing temperature and concentration of second phase particles (Al<sub>2</sub>O<sub>3</sub> particles) have significant influence on the hardness characteristics of electroless Ni–P–Al<sub>2</sub>O<sub>3</sub> composite coating.

To develop the new bath solutions with long cycles and high stability for electroless nickel (EN) plating of magnesium alloys, effect of bath pH and pH stabilizers on plating rate and coating quality as well as bath life were studied in detail by **Bonian Hu, Ruixue Sun, Gang Yu, Lingsong Liu, Zhihui Xie, Xiaomei He Xueyuan Zhang [10]**. Gravimetry was used to determine the deposition rate of coating. The coverage of coating was evaluated by 2 h of immersion test for magnesium alloys in 3.5 wt.% NaCl solution.

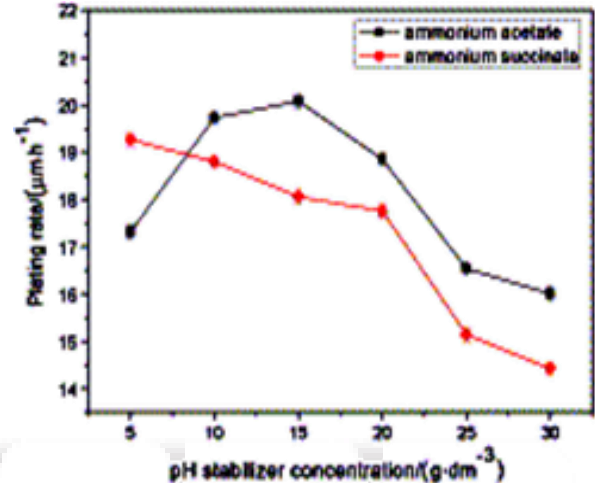


Fig. 5: Effect of pH on Plating ratio

The results showed that the bath pH not only changed the reactivity of the bath, but also had a strong impact on the microstructure and electrochemical properties of coatings. A highly uniform and dense coating was formed via EN plating in the new bath containing ammonium acetate. The bath life was extended when appropriate amount of ammonium acetate was added. It was effective for ammonium acetate to stabilize pH in the plating solution, to control coating quality and bath life.

### III. MATERIALS AND METHODS

A square M.S. plate of 50 x 50 mm has been selected. First the surface has been prepared with surface grinding and polishing with emery paper followed by buffing process.

The plate then been cleaned by degreasing, rinsing, drying. Acid pickling with 30 % concentrated HCl was carried out and then the surface was activated by dipping in 10 % concentrated H<sub>2</sub>SO<sub>4</sub>.

#### A. Bath Preparation:

Acidic bath is preferred as MS plate has to be plated.

Nickle sulphate	35 g/l
Sodium hypophosphite	25 g/l
Succinic acid	20 g/l
Lead acetate	0.0015 g/l
Acetic acid	30 ml/l
Thiourea	Stabilizer
Caustic soda	pH regulator

Sulphate alcohols	Wetting Agent
Distilled water	800 ml

The bath composition is as follows:

**B. Procedure Adopted:**

In an aluminium tank water is filled. In two beakers Electroless nickel bath and distilled water has been take.

Both beakers are then immersed in aluminium tank.

Level in Electroless Nickel bath beaker is marked so as to replenish the bath during plating.

Aluminium tank is heated with heater. The Electroless Nickel bath is indirectly heated through water media. As the temperature reaches to 60<sup>0</sup> C, M.S. plate was dipped into the bath for different period of time and the plating thickness had been noted.

Nickel metal concentration has been maintained between 5.3 to 6.0 g/l to obtain an optimum plating rate.

**C. Operating Conditions:**

- Temperature : 86 to 91<sup>0</sup>C (Optimum 88<sup>0</sup> C)
- pH 4.5 to 5.0
- nickel concentration 5.5-6.0 g/l
- work load 1.25 – 2.5 dm<sup>2</sup>/litre

**D. Observations:**

- (a) Weight of plate before plating: 19.6245 gm
- (b) Weight of plate after plating: 20.070 gm
- (c) Area of plate : 2500 mm<sup>2</sup>
- (d) Density of Electroless nickel : 8.91 gm/cc

Hence, the thickness of plating  
= Difference in weight / area x density

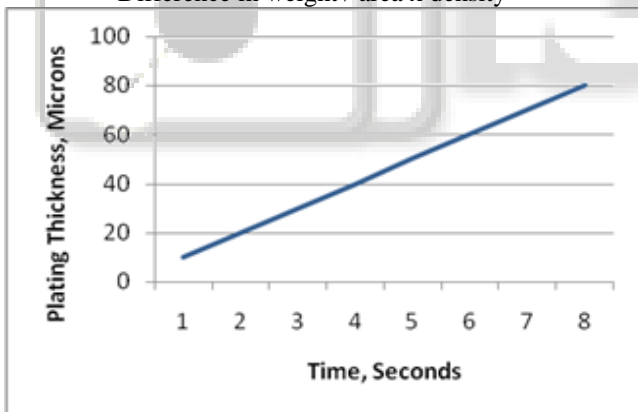


Fig. 6: Time vs Plating Thickness Graph

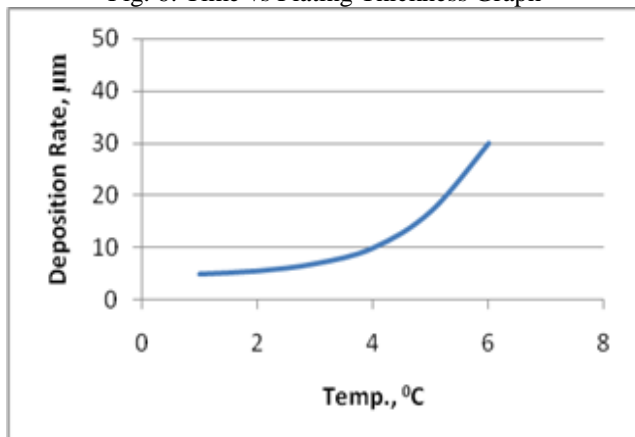


Fig. 7: Temperature vs Deposition Rate Graph

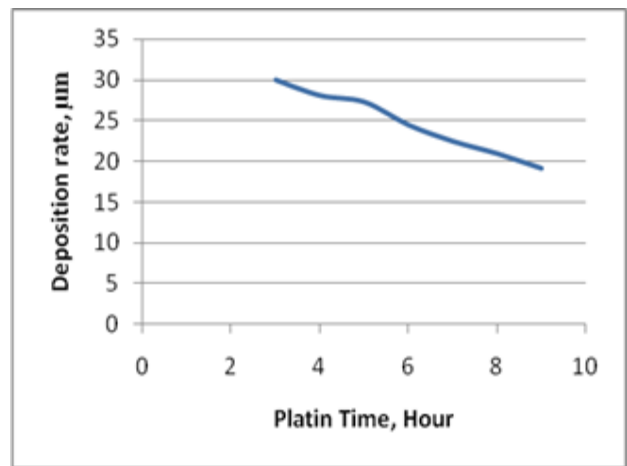


Fig. 8: Plating Time vs Deposition rate Graph

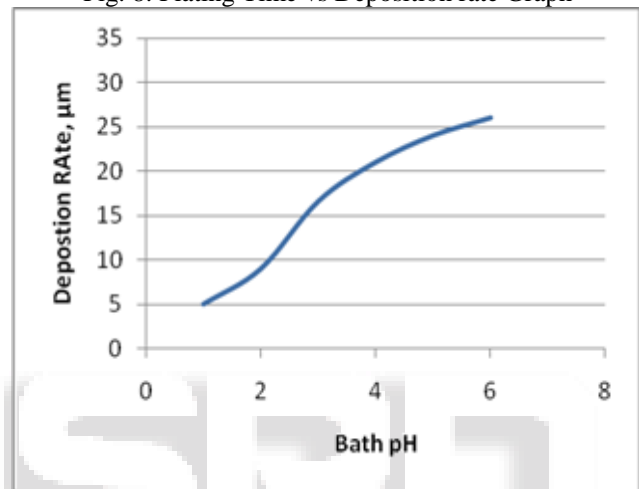


Fig. 9: Bath pH vs Deposition Rate Graph

**IV. RESULTS AND CONCLUSIONS**

For 30 minutes of immersion plating thickness = 20 µm and for 60 minutes of immersion, plating thickness of 40 µm had been found.

Nickel concentration has been calculated as 5.8gm/litre.

The deposition of nickel on steel surface is found to be influenced by temperature of bath, pH of bath, immersion time of specimen in the bath and on concentration of bath critically as shown in figures ,7,8,9 and 10.

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