Electroless Deposition and Characterisation of Nickel coatings

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ABSTRACT

Electroless Nickel plating is an Auto-catalytic reaction used to deposit, uniform and less porous Nickel-phosphorus or Nickel-boron alloy on a metal or plastic. Nickel coatings behave as proper barrier coating, protecting the substrate by sealing it off from the corrosive environment. Nickel coatings have excellent corrosion and wear resistance properties. This paper presents the experimental results of investigation of Electroless deposition and characterisation of Nickel coatings as Corrosion resistant coatings on Mild steel substrate. To observe the corrosion resistance behaviour of deposited nickel, Electrochemical Polarisation studies were carried out on nickel deposited Mild steel specimens in 5% NaCl solution. The Surface morphology of deposited nickel films were characterised by Scanning Electron Microscopic (SEM) technique. In polarisation study, these nickel coatings show good corrosion resistance towards 5% NaCl solution.

Keywords: *Electroless deposition, Nickel coatings, Corrosion resistance.*

INTRODUCTION

Electroless plating has been considered as an effective approach to provide protection and enhancement for metallic materials with many excellent properties like, hardness and wear resistance in engineering field. In metal finishing industries, electroless nickel plating is employed for many metal substrates including steel, copper, nickel, aluminium and alloys thereof. Generally, electroless nickel plating solution comprises a water soluble Nickel salt as metal source, a Complexing agent to increase the solubility of phosphite and to prevent nickel ion precipitation in solution, a Reducing agent in the form of hypophosphite or an amine borane and an acid or hydroxide, pH adjustor to provide required pH [1, 2]. The most common form of Electroless nickel plating produces a Nickel-Phosphorus alloy coating. The Phosphorus content has a great effect on deposit properties and it can be varied over a wide range, typically 3 to 12%. Higher phosphorus alloys are more corrosion resistant than those with lower phosphorus concentration. At low bath (acidic) pH values the resulting film will have increased phosphorus content. The factors that set the conditions for the corrosion resistance of electroless Nickel are: Surface properties and finish, Surface pre-treatment, Deposit thickness and Nature of the corrosive medium [3].

This paper reports the Electroless deposition and characterisation of Nickel films as Corrosion resistant coatings for Mild steel substrates like, nuts, bolts, washers and channels. The results on coating thickness, percentage composition of Nickel & Phosphorus, surface morphology and Corrosion resistance behaviour of deposited nickel films on mild steel substrate are included.

Experimental Procedure

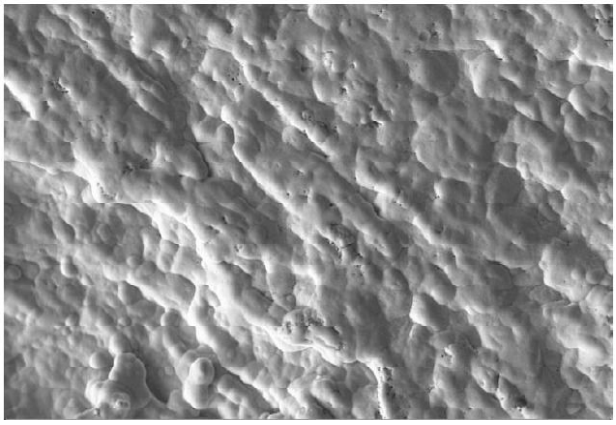
Plating solutions for Electroless deposition of nickel were prepared from Reagent grade chemicals and distilled water. Experiments were carried out using a Polypropylene plating tank having solution heating system, solution circulation with filtration system and mechanical agitation system. Nickel coatings were deposited using Electroless deposition technique on suitably pre-treated mild steel panels (Solvent degreasing, acid pickling and bright dip) at 85deg C in a plating solution at pH 4 to 5 under stirred condition. Nickel films grown using acidic baths exhibit good adhesion characteristics to steel. Deposition time was varied to study the effect on

coating thickness build-up. The Corrosion behaviour of deposited nickel films were studied by Electrochemical Polarisation technique at 298K in 5% NaCl solution. Electrochemical methods are popular because they can be carried out in a few hours. They are based on the measurement of the current as a function of the voltage applied to the test specimens. Polarisation diagram is constructed using current and potential values and from the diagram, the Electrochemical Polarisation data for Corrosion kinetics on Nickel coated specimens are derived. In Polarisation technique, a Platinum coil and saturated Calomel electrode were used as Auxilliary and Reference electrode respectively. The Surface morphology of the deposited nickel coatings were studied using Scanning Electron Microscopic (SEM) technique. SEM is a type of Electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the sample's surface topography and composition. Thickness and Phosphorus content of nickel coatings were measured using X-ray fluorescence (XRF) equipment. XRF is a non-destructive analytical technique used to determine the elemental composition of materials and coating thickness. XRF analyzers determine the chemistry of a sample by measuring the fluorescent (or secondary) X-ray emitted from a sample when it is excited by a primary X-ray source.

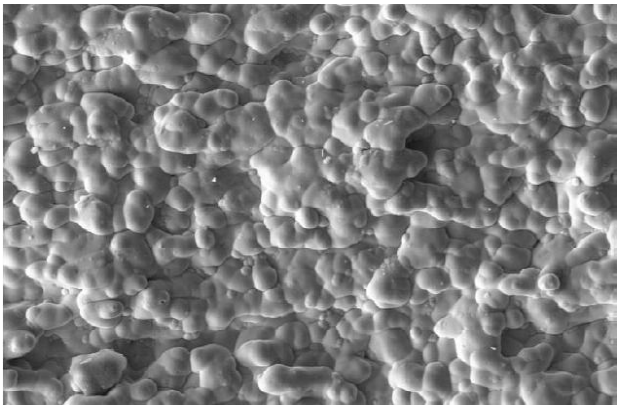
Results and Discussion

Nickel coatings were deposited using Electroless plating solution at different deposition time (1hr to 4hrs) using bath solution containing $NiSO_4$ $6H_2O$ - 45g/l, Glycine - 40g/l, Acetic acid - 10ml/l and Sodium hypophosphite - 10g/l. Prior to Electroless deposition, mild steel specimens were cleaned and prepared. Deposition time was varied to achieve 20 to 25µm thick, corrosion resistant nickel coating.

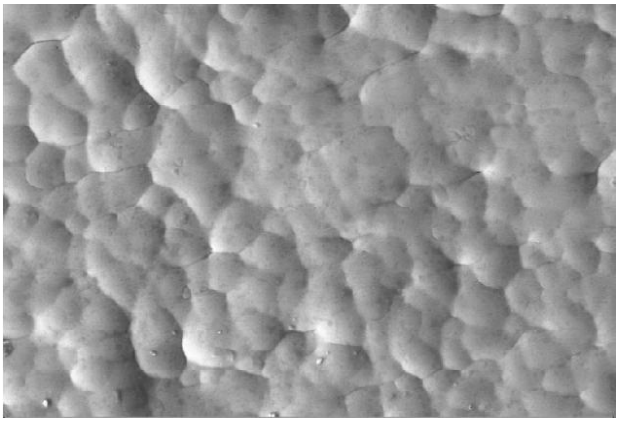
Scanning Electron Micrographs (SEM- Zeiss EVOMA 15 3004980) of nickel films coated at different deposition time at 400X magnification are shown in Figure. 1. The Nickel films coated at 1hr and 2hrs deposition time, exhibit rough surface with smaller grains (Fig. 1a & 1b). Whereas, Nickel films coated at 3hrs and 4hrs deposition time show smooth surface with larger grains (Fig. 1c & 1d).



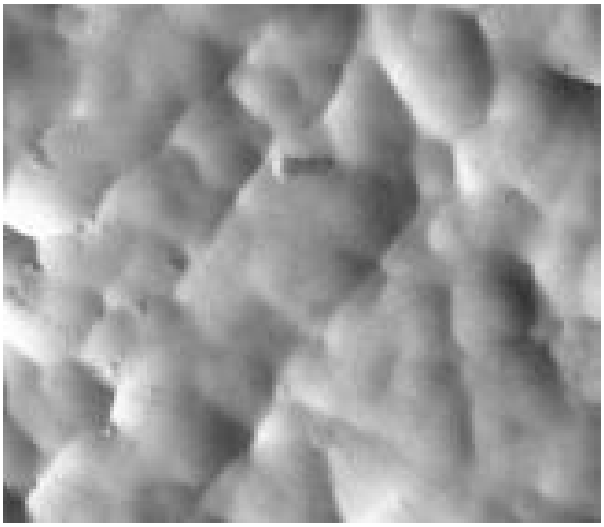
(a)



(b)



(c)



(d)

Fig. 1. SEM Photographs of Nickel films coated at different deposition time (hrs)

(a) 1hr, (b) 2hrs, (c) 3hrs and (d) 4hrs

Figure 2. shows the Effect of Deposition time on coating thickness. It shows coating thickness increases with increase in deposition time and the required coating thickness (20 to 25µm) was obtained in 4hrs deposition time. Hence, maximum deposition time was limited to 4hrs duration. Plating solution has been regularly replenished by adding nickel sulphate and sodium hypophosphite to achieve required coating thickness.

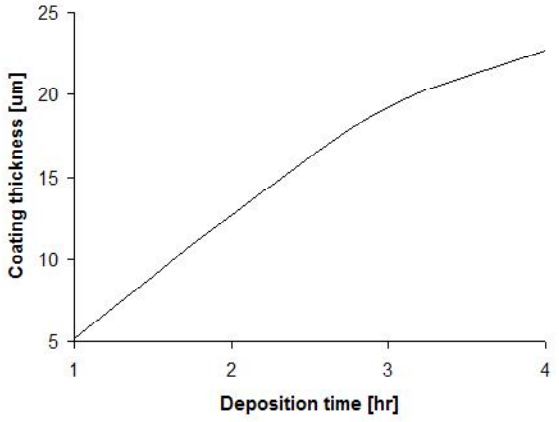


Fig. 2. Effect of Deposition time on coating thickness

Since, Phosphorus content in nickel coatings has a great effect in enhancing corrosion resistance property, percentage composition of Phosphorus was measured. Table 1. gives the Nickel and Phosphorus content in deposited nickel films. It shows, all coated nickel films at varying Deposition time, have resulted high phosphorus content.

Table 1

Percentage composition of Nickel and Phosphorus in deposited nickel coatings.

Deposition time(hr)	Nickel content(%)	Phosphorus content(%)
1	87.98	12.02
2	89.41	10.59
3	88.89	11.11
4	90.04	9.960

Electrochemical Polarisation experiments were conducted in 5% NaCl solution on Electroless nickel plated mildsteel specimens of area 1cm^2 by sweeping potential (±250mV) both Anodically and Cathodically from its open circuit potential (OCP) at 298K. The current response w.r.t applied voltage was studied and the Electrochemical Polarisation data derived (Table 2). The Electrochemical Polarisation data derived from the Polarisation studies indicates, that the Corrosion resistance behaviour of Nickel coated Mild steel specimens increases with increase in deposition time by showing decreasing values of i_{corr}, mpy and gram/hr. Hence, the performance of nickel coatings increases with increase in Deposition time (with increase in coating thickness) and the nickel film deposited for 4hrs duration, performs as a better corrosion resistant coating with lower i_{corr} value than the value obtained with other systems (Nickel coatings deposited for 1hr, 2hrs, 3hrs deposition time and bare mild steel substrate).

Table 2

Electrochemical parameters for Corrosion kinetics on Nickel deposited mild steel specimens in 5% NaCl medium at 298K

Deposition time[hr]	**i_{corr} [µA/cm^2]**	**[mpy]**	**[gram/hr]**
Mild steel substrate	3.655 x10^2	1.668 x10^2	3.808 x10^{-4}
1	9.665	4.412	1.007 x10^{-5}
2	4.945	2.257	5.152 x10^{-6}
3	4.209	1.921	4.385 x10^{-6}
4	2.852	1.301	2.971 x10^{-6}

Conclusion

Electroless plating bath composition and surface preparation procedure prior to plating have been optimised to obtain uniform, adherent and corrosion resistant nickel coatings. Deposition time was varied from 1hr to 4hrs duration. Since, the required corrosion resistant, 20 to 25micron thick nickel coating was obtained for 4hrs deposition time, the maximum deposition time was optimised as 4hrs duration. These deposits were studied for its surface morphology, coating thickness and Phosphorus content. Nickel deposits are characterised as effective corrosion resistant coatings for Mild steel substrates through Electrochemical Polarisation method in 5% NaCl medium at 298K. The corrosion resistance value of nickel coatings increases with increase in deposition time by showing decreasing value of i_{corr}. The nickel coating which is deposited for 4hrs duration, has shown smooth surface with uniform deposition and exhibited better performance as corrosion resistant coating for mild steel material by showing lower value of i_{corr}.

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