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Evaluation of Corrosion Properties of Heat Treated Nickel-Phosphorus Electroless Coatings

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Abstract—*Electroless Nickel-Phosphorus (NiP) composite coatings on Mild Steel (MS) have demonstrated significant improved mechanical and tribological properties. The corrosion resistance of these composite coatings, however has not been systematically studied and compared. Our present work aimed at evaluating corrosion characteristics of heat treated NiP coatings using standard corrosion tests which include Immersion Corrosion Weight Loss Method and Salt Spray Corrosion Test. The effects of the co-deposited particles on corrosion behavior of coatings in 3% and 5% NaCl media were investigated. The effect of heat treatment on coating corrosion resistance was also discussed. The results showed that NiP composite coatings demonstrated significant improvement of corrosion resistance in both the tests. It was understood that thermal effect significantly improves the coating density and structure, giving rise to enhanced corrosion resistance.*

Keywords*: Coatings, Corrosion, Heat Treatment, Ni-P composites*

1. INTRODUCTION

Corrosion inhibition has been a topic of interest of researchers **for** decades [1-2]. Electroless Nickel coatings are known to resist corrosion and have been widely used in chemical and mechanical industries [3]. They are known to protect the substrate by covering it off from the corrosive environment. Electroless NiP coating is an autocatalytic chemical reduction process in which the reducing agent is oxidised and Ni^{+2} ions are deposited on the surface. Once the first layer of Ni is deposited, it acts as a catalyst for the process [4]. However, the drawback of this process is that any pores or cracks in the coatings make it ineffective. Therefore the absence of pores and cracks is imperative for a good corrosion resistant NiP coating. Heat treatment can cause significant changes in the properties and structure of NiP coatings [5]. It is reported that heating of NiP deposits at 573-673 K can cause significant improvement in their hardness and wear resistance [6-7] which in turn leads to an improved corrosion resistance [8]. Our present work aims at investigating the corrosion resistance properties of NiP coatings and to study the effect of heat treatment on corrosion resistance. Weight Loss Corrosion Test and Salt Spray Corrosion Tests were performed to evaluate the corrosion resistance of heat treated and bare samples.

2. MATERIALS AND METHODS

2.1 Synthesis of NiP coatings

An electroless Nickel bath was chosen as coating bath. Nickel Chloride hexahydrate was used as a source of Nickel while Sodium hypophosphite served as the reducing agent and source of Phosphorus. Besides these, the bath also contained suitable amounts of complexing agents and stabilizers. Table 1 shows the chemicals used and their role in the reaction. The bath was operated at a temperature of 80°C with continuous stirring. Mild Steel (MS) samples (40 mm x 15 mm x 3 mm) having a composition of C 0.16%, Si 0.4%, Mn 0.7%, S 0.04% and Fe (balance) were used as substrate materials. Fig 1. shows the CAD model of the substrate. The pieces were surface ground and thoroughly washed with deionised (DI) water. They were subsequently cleaned by dipping in 1M HCl for five minutes followed by washing with DI water.

Table 1: List of chemicals and their role in electroless coating

Sl. No.	Chemicals	Role of Chemicals
01	Nickel chloride	Provide metal ions for deposition.
02	Glycene	Complexing and buffering agent.
03	Sodium hypophosphite	Reducing agent.
04	Sodium acetate	Accelerator.
05	Sodium hydroxide	To facilitate Exothermic reaction.

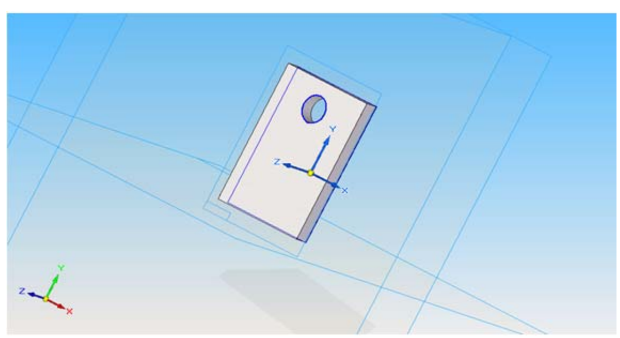

Fig. 1.a CAD model of a flat specimen (MS Substrate)

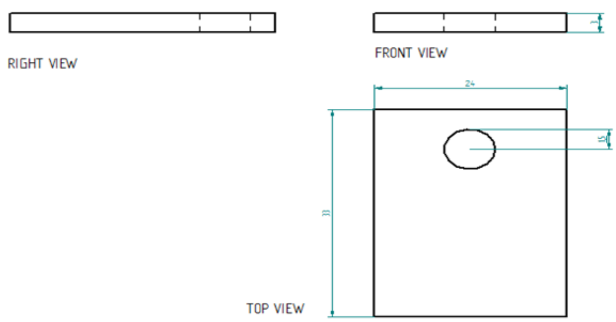

Fig. 1.b 2D drawing of a flat specimen (MS Substrate)

The prepared substrates were tied to a copper string and dipped in the coating solution for 1 hour with uniform stirring. After coating, the samples were thoroughly washed with the DI water and dried.

2.2 Heat Treatment

Heat Treatment was carried out in a typical heat treatment furnace. The specimens were wrapped with aluminum foil to prevent surface oxidation and phosphorus depletion. The coated samples were heat treated at 600°C for 1 hour and cooled in different atmosphere (furnace, ice and ambient air) to study the effect of heat treatment on coatings.

2.3 Corrosion resistance measurements

2.3.1 Immersion Corrosion Weight Loss Method.

The coated and heat treated samples were weighed and immersed in a 3% NaCl solution and allowed to corrode for one week and the samples were weighed again after the test. The rate of corrosion was calculated through the difference in the weight of the sample.

2.3.2 Salt Spray Corrosion Test.

The apparatus for a Salt Spray Corrosion Test consists of a closed testing chamber, where a salt solution (5% NaCl) is atomized by means of a nozzle. This provides a corrosive environment of sodium chloride fog such that the samples are subjected to severe corrosive conditions. The test was carried out for one week in a single run. The coating loss was determined as a function of time lapse.

3. RESULTS AND DISCUSSIONS

Corrosion resistance of the coated and heat treated samples was compared with that of the samples that were coated but not heat treated. Immersion Corrosion Test showed a maximum weight loss in the sample that was not coated (a) to the one that was coated but not heat treated (b). Weight loss was minimal in the sample that was coated, heat treated and cooled in the furnace environment (e). Heat treated and ice cooled (c) and air cooled (d). Samples showed intermediate weight loss in that order. Fig. 2 shows the graphical representation of weight loss obtained through Immersion Corrosion Test.

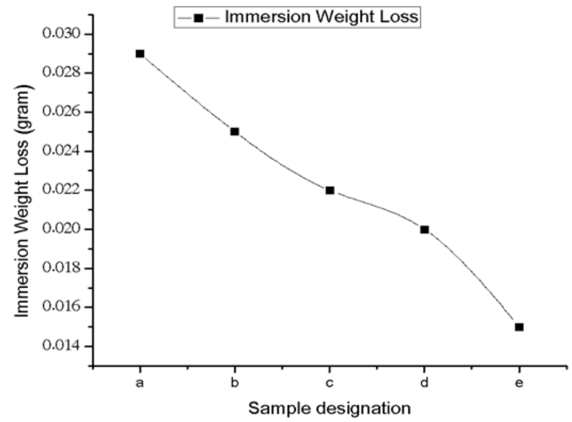

Fig. 2: Weight loss in Immersion Corrosion Test

Salt Spray Corrosion Test showed similar results in terms of weight loss of the sample as shown in Fig. 3

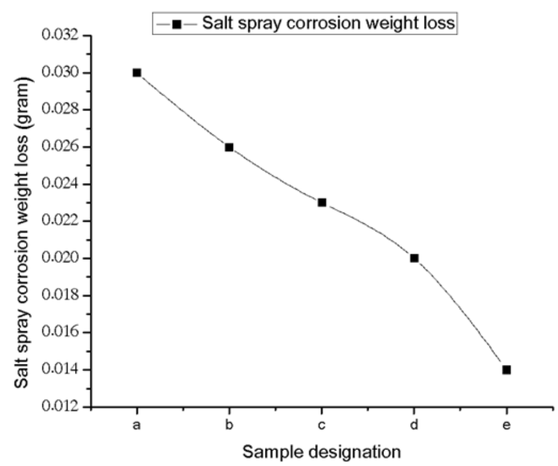

Fig. 3: Weight loss in Salt Spray Corrosion Test

From both the corrosion tests, it was observed that, coated and heat treated samples subjected to uniform cooling showed improved corrosion resistance. Energy Dispersive X-Ray Analysis (EDAX) confirmed the presence of electroless coatings on the sample as shown in Fig. 4.

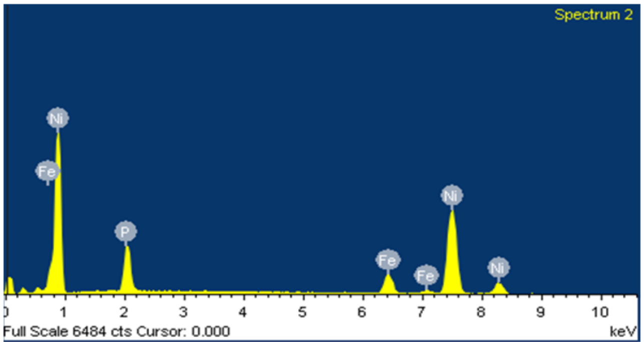

Fig. 4: EDAX of electroless NiP coated MS substrate

It is known that heat treatment can cause significant changes in the structure and properties of electroless Ni-P coatings [5]. X-Ray Diffraction Analysis (XRD) was performed to understand the effect of heat treatment in the improvised corrosion resistance. Fig. 5 shows the X-Ray diffractogram of the electroless NiP coating before heat treatment. The X-ray pattern shows a typical diffraction pattern reported for amorphous electroless NiP coatings [9]. Heat treatment enabled the crystallization of NiP, stabilizing the coating. Heat treated and furnace cooled sample was uniformly cooled and showed better performance characteristics.

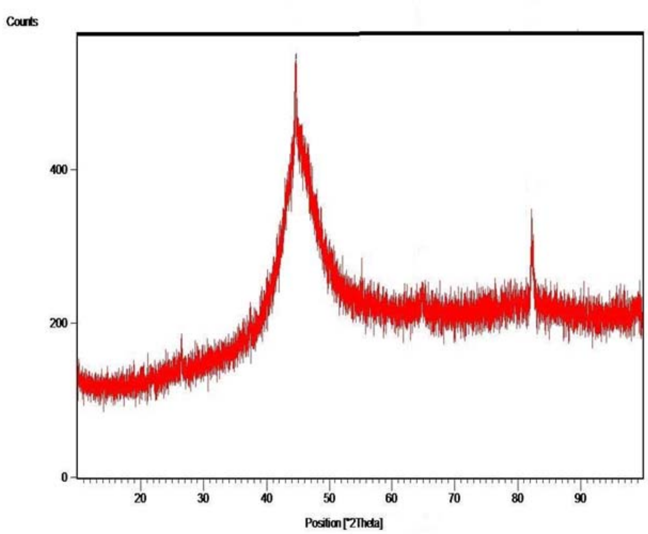

Fig. 5: X-Ray Diffractogram of NiP coated substrate

4. CONCLUSION

Heat treatment causes significant improvement in the corrosion resistance of electroless coatings. Rate of cooling plays a vital role in the improvisation of performance characteristics. Studying the heat treatment effect on electroless Ni-P coatings in different cooling media will be our further interest.

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