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INFLUENCE OF ELECTROLESS NICKEL-PHOSPHORUS DEPOSITION ON HARDNESS OF MILD STEEL AND AZ91 MAGNESIUM ALLOY

MOHD IMRAN ANSARI, D. G. THAKUR

Mechanical Engineering Dept, DIAT (DU), Pune, India

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Abstract: Electroless nickel coating (ENC) has many advantages in electronic, automobile, aviation industries, due to their good resistance to wear and corrosion, uniformity of deposit and ability to impart high hardness. Electroless deposition is an autocatalytic method used to deposit on a substrate in the presence of a reducing agent. Electroless Ni-P coatings were applied to the mild steel and AZ91 magnesium alloy for improving its micro-hardness. In the present investigation, the effect of Ni-P depositions on mild steel and AZ91 alloy resulted in smooth microstructure of the substrate surface and improved micro-hardness. Post heat treatment hardness values increase in MS and decrease values in AZ91 alloy. The microstructural and phase structure analyses were conducted with the help of field emission scanning electron microscopy (FESEM), energy-dispersive X-Ray (EDX) analysis and X-Ray diffraction (XRD) analyzer, Atomic Force Microscopy (AFM) respectively.

Keywords: EN coating; AFM; Micro-hardness

Corresponding Author: MR. MOHD IMRAN ANSARI

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INTRODUCTION

Electroless Ni-P coating alloy is well established surface engineering process that involves deposition of a metal alloy coating on various substrates. Although a variety of metals can be as plated, it has received widespread acceptance as it provides high hardness and excellent resistance to wear, corrosion and abrasion (Gordani, et al 2008).

Electroless nickel-phosphorus deposition is established industrial practice as a protective and decorative coating in various industries due to its superior corrosion and wear resistance, excellent uniformity, wide range of thickness as well as mechanical and physical properties. More research has carried out on the characterization of electroless nickel coating process. Temperature, pH, nickel ion concentration, reducing agent, complexing agent etc. concluded that nickel ion is catalytic reduced by means of the active atomic hydrogen with immediate formation of orthophosphate and hydrogen ions (Gutzeit, et al, 1959). The electroless nickel deposition bath is known to have a major problem of sudden bath decomposition, so that results rising the operating cost also the generation of environmental hazardous waste (Cheonga, et al, 2004). Electroless nickel plating on magnesium alloys is a proper protection method because it is environmentally-friendly and the hardness of EN deposits can be increased by heat treatment due to the formation of nickel phosphide (Ni3P) (Sankara, et al, 2003). Maximum Ni3P is formed when annealed at 4000 C for 1 hour (Sudagar, et al, 2013). Hence, the present study focuses on the refinement in microstructure of electroless Ni-P alloy deposit on the substrates. Deposit structure and surface morphology have been characterized by XRD, field emission scanning electron microscopy (FESEM) and atomic force microscopy (AFM). Micro-hardness measurements have been made on these deposits with and without heat treatment.

MATERIALS AND METHODS

Experimental Setup

Electroless Ni-P coating is deposited on mild steel and AZ91 magnesium alloy substrate of size (20X20X1.5 mm). A pin-hole was drilled at the corner for ease of suspension in the chemical bath. The following surface preparation of the substrate was carried out prior to conduct of the experiment: (i) mild steel specimen was mechanically cleaned for removal of corrosion, degreased in acetone, cleaned with a NaOH solution, etched in 10 ml of 40 percent by volume HCl for 02 minutes and cleaned with 50 gm/l NaH2PO2 for 10 minutes with rinsed in distilled water between consecutive steps. (ii) AZ91 magnesium alloy (Al 9.2%, Zn 0.84%, Mn 0.28%, bal. Mg (wt%)) specimen was mechanically cleaned for removal of corrosion, degreased in acetone, rinsed in 10% NaOH at 600 C for 05 minutes, 6% chromic acid - 5% nitric acid pickling for 45 sec., followed by fluoride activation in HF (250 ml 70% HF per litre) for 10 min. With rinsed in distilled water between consecutive steps. Specimens were then transferred immediately to the Ni-P coating solutions. Before and after immersion, the specimens were weighed using a Mettle analytical balance. Finally, the specimens were put into the electroless solution for coating. After plating, again the specimens were rinsed in distilled water, dried and preserved for characterization. The compositions of bath used and operating conditions of electroless Ni-P coatings on mild steel (M.S) and AZ91 Mg alloy were as given in Table 1.

Table 1. Bath compositions and operating conditions of electroless Ni-P coatings

Bath constituents	Concentration	
	M.S	AZ91 Mg
Nickel sulphate hexahydrate (g/l)	20	20
Sodium hypophosphite monohydrate (g/l)	24	24
Citric acid monohydrate (g/l)	6	6
Lead acetate (g/l)	0.003	0.003
Ammonium bifluoride (g/l)	-	10
Hydrofluoric acid, HF (40% V/V)	-	12
Operating conditions		
pH	8	8
Temperature (^{0}C)	85±1	85±1

Experimental Methods

EN deposition was carried out on a spinot hotplate with magnetic stirrer. The bath was operated for duration of 60 mins. The coating thickness of the deposit was calculated using the formula mentioned below and verified using a Coating thickness gauge:-

$t = (W2-W1) / \rho * A * T$ (1)

Where,

t is the coating thickness (cms),

W1 is the weight of substrate before coating (gms),

W2 is the weight of substrate before coating (gms),

ρ is the density of the deposit (gms/cm3), A is the surface area exposed to coating (cm2),

T is the deposition time in hours.

The micro-hardness (Vickers Hardness) was measured using Tukon 1202 micro hardness tester. A load of 500 gm was applied for a dwell time of 10 seconds to obtain the indent. The hardness was measured on the as-coated samples and again post heat treatment to analyse the effect heat treatment on the electroless Ni-P deposits.

Selection of Electroless Ni-P Coating Bath

The phosphorus content can be controlled easily in due respect of their properties. On the basis of phosphorus content the deposits can be sub-classified into Low (3-5 % P), Medium (6-9 % P) and High (10-14 % P) Phosphorus (Sudagar J. et al., 2003).

RESULTS AND DISCUSSION

Surface Morphology, Composition and Structure

Electroless Ni-medium and high Phosphorus deposits obtained using baths (Table 1) respectively, are uniform and highly adherent. Fig. 1 shows FESEM (Field Emission Scanning Electron Microscopy) [Model: Σigma, Carl Ziess] morphology and EDX (Energy Dispersive X-Ray) analysis strongly suggest the presence of phosphorus in the coating. These figures reveal that the deposit possess a globule type structure, as well as good homogeneity and high density.

The X-Ray diffraction [Model Name: Bruker AXS, Germany (D8 Advanced)] pattern of electroless Ni-P deposits, obtained by baths in their as plated conditions, are given fig. 2. In all the diffraction patterns, the reflections corresponding to the (111) plane of a face-centred cubic (fcc) phase of nickel could be observed. It is evident from fig. 2 and that as-deposited Ni-P deposit had only a single broad peak at 44.80 2θ. Literature review on Ni-P coatings has shown that Ni-P deposits having greater than 7 wt. % phosphorus are amorphous (Mai Q. X.). The elelctroless deposit on AZ91 Mg alloy is amorphous in nature whereas that on the mild steel is microcrystalline in nature, as inferred from the XRD pattern (fig. 2).

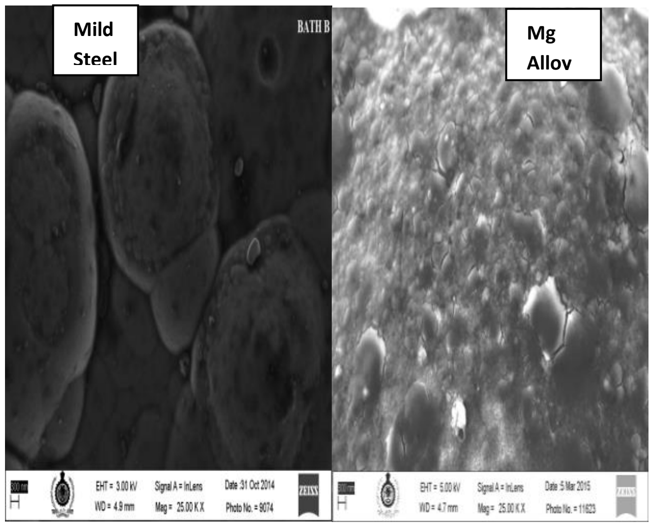

Fig. 1 SEM micrographs of as-plated of Ni-P deposit on MS and AZ91 Mg alloy

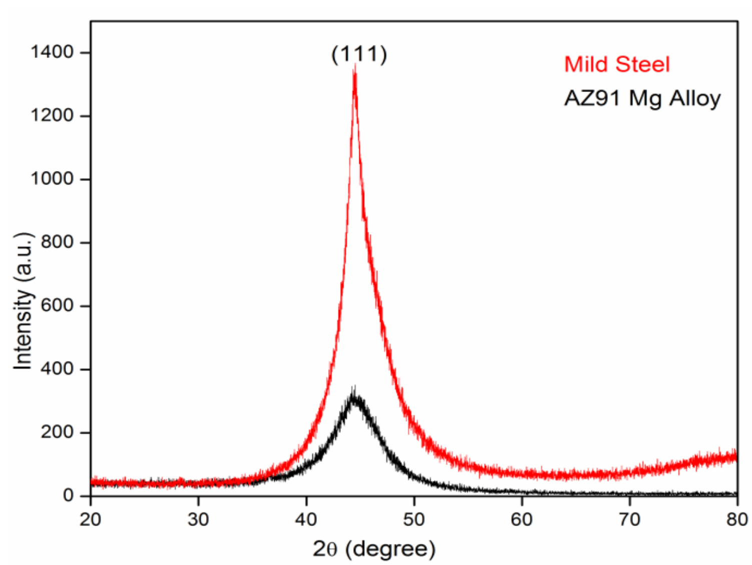

Fig. 2 XRD patterns of as-deposited electroless Ni-P coating

Figs. 3 and 4 show the three dimensional AFM images for the as-deposited electroless nickel-phosphorus alloy for duration 60 sec on mild steel and AZ91 Mg alloy. These substrates have an initial roughness of 12 nm. By comparing the images, it is clear that the deposits have different surface roughness values due to different growth pattern of the deposit. The average roughness value obtained for Ni-P deposit of Mild Steel is 17.5 nm and AZ91 Mg alloy is 43.9 nm.

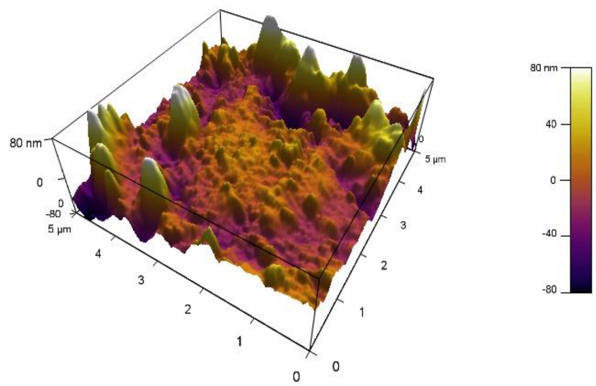

Fig. 3 AFM morphology of the as-plated ENC of MS substrate of Ni-P alloy

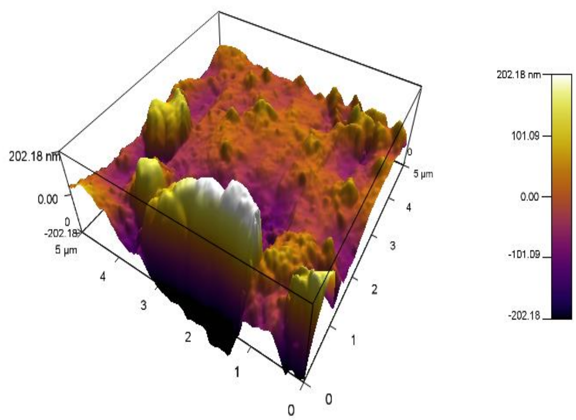

Fig. 4 AFM morphology of the as plated ENC of AZ91 Mg alloy of Ni-P alloy

Table 2. Micro hardness of electroless Ni-P deposits in as-plated and heat-treated conditions

Ni-P deposits on substrate	Phosphorus content (wt%)	Coating Rate (µm/h)	Micro-hardness (HV_{500})		
				Heat-treated	
	As-plated			200 °C	400 °C
Mild Steel	13	23	295	307	346
AZ91 Mg alloy	10	101	270	336	225

CONCLUSION

Based on the experimental results and analysis, the following conclusions have been drawn which clearly indicates the significant improvement of the hardness performance on heat treatment.

On heat treatment of the coated Mild Steel substrate at 4000 C the hardness was observed to increase from 295 to 346. This increase in hardness may be attributed to increase in grain growth.

It was observed that the rate of coating (µm/h) on AZ91 Mg alloy is higher than Mild steel.

FESEM study shows that coatings are smooth, uniform and low porosity. The XRD analysis shows that the films contain Nickel as primary component of the coating. Electroless Ni-P coatings have been found to have high corrosion resistance, abrasion resistance and hence are suitable for marine applications.

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