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Effect of heat treatment on mechanical and tribological characteristics of Electroless Ni-P deposits

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Abstract. In the present paper, mechanical and tribological characteristics of as-deposited and heat-treated Electroless Ni-P coatings were investigated. Medium phosphorus Ni-P coatings were deposited on mild steel substrate using an acidic bath and subjected to heat treatment at 400°C in a tubular furnace. Surface morphology and elemental analysis of the coatings were carried out using Scanning electron microscope (SEM) and Energy dispersive X-ray spectroscopy (EDS) analysis respectively. Dry sliding wear characteristics of the coatings were investigated by linear reciprocating ball on flat tribometer using AISI E52100 Steel ball as a counter body at room $(25\pm2^{\circ}C)$ and elevated temperature $(300^{\circ}C)$. Hardness results showed significant improvement in the surface hardness of coatings on heat treatment. Low friction coefficient and high wear rate were observed at elevated temperature in comparison of those of tests conducted at room temperature for both as-deposited and heat-treated coatings. Best tribological performance is observed for heat-treated coating tested at room temperature.

1. Introduction

Electroless nickel coating is an alloy of nickel and phosphorus. As the name suggest it does not require electricity for operation, unlike traditional electroplating process which requires external source of current to drive the deposition reaction. Instead, it is an autocatalytic reduction of metallic ions (Ni^{2+}) from an aqueous solution containing a reducing agent. The driving potential of the deposition reaction is provided by the reducing agent and the temperature of the plating bath [1]. Once the first layer of Ni is deposited, it acts as a catalyst for the subsequent deposition reactions. Electroless nickel plating can be divided into three main types: low phosphorus (3 to 5wt % P), mid phosphorus (6 to 9 wt% P), and high phosphorus (>10 wt% P) [2].

Heat treatment of Ni-P coatings improves mechanical and wear characteristics of the coating [3]. The optimum temperature for heat treatment was found to be around 400°C for 1 hour, at this temperature hardness was found to be maximum [4]. This increase in hardness can be attributed to formation and precipitation of hard Ni₃P phase. There are several studies on tribological behaviour of Ni-P coatings at elevated testing temperatures with pin on disc configuration [5]. Review of literature indicated that very few researchers studied the tribological behaviour of the Ni-P coatings at elevated temperatures using Ball-on-plate configuration.

The main objective of this paper is to study effect of heat treatment on hardness and tribological characteristics of the medium phosphorus Ni-P coatings at room and elevated testing temperatures under high contact stresses against AISI E52100 steel ball.

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2. Experimental details

2.1. Substrate activation and coating deposition

Mild Steel specimens were used to successfully deposit electroless nickel coatings. Square specimen of 20 mm edge length and 5 mm thickness are cut from Mild Steel plate. The plate is parted, drilled for sample holding and then surface grinded using SiC emery paper of grades 100, 150, 220, 320, 600, 800 in successive order to achieve sufficient surface finish. Then the substrate undergoes 20 minutes of ultrasonic cleaning using acetone solution followed by 30 minutes of alkaline cleaning using 10% Sodium hydroxide at $60\pm5^{\circ}$ C. To activate substrate catalytically for the coating process, acid pickling in the 20% by volume HCl for 2 minutes is done. Then the sample is rinsed in distilled water before dipping in to the electroless nickel bath.

The bath composition plays an important role in determining the properties of the coating. The electroless bath is basically a blend of chemicals including Nickel source, a reducing agent, complexants, stabilizer, and surfactant. Electroless nickel solution containing 5-6 g/L of nickel metal, 27-33 g/L of sodium hypophosphite and other proprietary additives was used as an electrolyte solution for depositing medium phosphorus electroless nickel coating. The coating process is initiated when a catalytically activated sample is immersed in the electroless nickel bath. The electroless nickel bath was indirectly heated in an oil medium (SAE 40) to the required temperature by using the Digital heater-cum-stirrer which also has a temperature sensor. The temperature of the oil medium is controlled and corresponding temperature of the plating solution was monitored using a mercury-glass thermometer. During the deposition process, the pH of the plating solution held at the desired range by adding suitable amounts of 50% liquor ammonia. Total time for the plating process is 150 minutes with initial volume of the plating solution of 150 ml and maintained throughout the process by replenishment. The bath agitation is very crucial to get quality deposits as it ensures the proper escape of hydrogen gas which otherwise would cause pitting of the surface. The magnetic stirrer with the stirring speed of 250 rpm ensures proper agitation. Bath composition and operating conditions are described in 'table 1'.

Operating parameter	Range
Nickel metal	5-6 g/l
Sodium hypophosphite	24-36 g/l
Temperature	85±2°C
pH	5.0-6.0
pH adjusters	50% Liquor Ammonia
Deposition time	2 hrs 30 minutes
Bath Volume	150 ml
Bath agitation (Magnetic stirring)	250 rpm

Table 1. Operating parameters for Ni-P coating deposition

Heat treatment of Ni-P alloy coatings can cause significant changes in properties and structure. Heat treatment of Ni-P samples was carried out in an air atmosphere in the tubular furnace (INDFURR superheat furnace) at 400°C for 1 hour [6].

In this study, the deposition thickness was calculated using the equation 1 the deposition rate was calculated based on weight gain method using the equation 2. The deposition rate calculated to be approximately $35 \pm 2 \ \mu m \ h^{-1}$.

Coating thickness =
$$\frac{W_1 - W_2}{\rho \times A} \times 10^4 \,\mu m$$
 (1)
Deposition rate = $\frac{W_1 - W_2}{\rho \times A \times T} \times 10^4 \,\mu m \,h^{-1}$ (2)

W₁, W₂ represents weights of sample before and after coating (g); ' ρ ' denotes density of nickel (g cm⁻³); 'A' denotes surface area of mild steel substrate (cm²); 'T' denotes coating time (hours)

2.2. Characterization studies on the coatings

The surface morphology and elemental composition of as-deposited and heat treated coatings were analyzed by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX) using JEOL JSM-638OA scanning electron microscope. Hardness has no direct connection to material strength, but it is a reliable indicator of the degree of abrasive-wear resistance. Hardness, in general, is affected by heat treatment. The micro-hardness of the as-deposited and heat-treated coatings was measured using MITUTOYO micro-hardness tester at a low load of 100g for a dwell time of 15s. The average of 5 readings taken at different positions was reported here.

DUCOM linear reciprocating tribometer TR-281 is used to investigate the friction and wear characteristics of as-deposited and heat-treated coatings under dry non-lubricated sliding conditions at room and 300°C operating temperatures. The specimen is held tightly in the specimen holder. A hardened steel ball of 6 mm diameter is pressed against the specimen under a fixed load of 10N. The ball undergoes back and forth sliding reciprocating motion under a fixed frequency and stroke length for a particular period of the time. To obtain statistical confidence the experiments were conducted on at least 3 samples of as-deposited and heat treatment coatings. The data is plotted by the data acquisition software (WINDUCOM 2010) installed in the workstation controlling the tribometer. The test conditions and operating parameters are listed in 'table 2'.

Specific wear rate of as-deposited and heat treated coatings at room and elevated temperature was calculated using the relation, $K = V/(S \times F)$ where V is wear volume (mm³) i.e., weight loss/density, S is sliding distance (m) and F is normal load (N).

Configuration	Ball on plate
Operating mode	Linear reciprocating
Counter Ball material and	AISI E52100 steel, 6
diameter	mm
Load	10 N
Stroke Length	5 mm
Frequency	3 Hz
Sliding distance	100 m
Atmosphere	26±2°C & 300°C

 Table 2. Operating conditions of Tribometer

3. Results & Discussion

3.1. Morphological observations and elemental analysis of the coatings

The surface topography is a significant factor that controls the friction and transfer layer formation during the sliding of two materials. 'Fig. 1' shows the surfaces of typical electroless Ni–P coatings in as-deposited condition. From surface morphology of Ni-P coatings shown in figure, it is evident that homogeneous fine globular structures with good uniformity and dense coverage are observed on the as-deposited coating. Non-porous and uniform profile is the characteristics of Ni-P coating evidenced in SEM image [7]. In general, the advantage of non-porous and absences of voids permit the coating to restrict from the corrosion of the samples. Heat treatment of the coatings at 400°C increases the size of the globules presenting coarse grained structure as observed in 'Fig 1(b)' [8].

EDS analysis of coatings revealed the elemental composition of as-deposited coating and heattreated coating. As-deposited coating consists of 7.32 wt% phosphorus content confirming the deposition of medium phosphorus electroless Nickel coating. Heat treatment at 400°C under air atmosphere increased the oxygen content (18.74 wt%) in the coating thereby confirming severe oxidation. The phosphorus percentage is found to be decreased to 3.80 wt%.



Figure 1. Surface morphology and corresponding EDS analysis of a) Ni-P as-deposited coating b) Ni-P heat treated coating

3.2. Hardness behavior of Ni-P coatings

Surface hardness is an important factor that affects the frictional and wear properties of the coatings. It is well known that Ni-P coatings are known for their high hardness. Further heat treatment temperature and duration has significant impact on the hardness of the coatings. In the present study heat treatment is done at 400°C for 1 h in air atmosphere. 'Fig. 2' shows the hardness of as-deposited and heat treated Ni-P coatings. The hardness of as-deposited and heat-treated coatings are measured to be 410 ± 30 HV and 820 ± 25 HV respectively. The increase in hardness in heat treated coatings is attributed to precipitation of Ni₃P phases. Hard Ni₃P phases acts as barrier for dislocation movement, this results in reduced plastic deformation [9-11].



Figure 2. Bar diagram showing the Vickers hardness of Ni-P coatings

3.3. Tribological behavior of the coatings

The friction coefficient generally increases with an increase in load and decrease in sliding velocity. Thus a combination of high load and low sliding velocity serves as a good operating condition to predict the durability of the coating. 'Fig. 3' illustrates the frictional behavior of asdeposited and heat-treated coating at room and 300°C testing temperatures against AISI E52100 steel ball. It is observed from 'Fig. 3(a)' that at room temperature the as-deposited coating shows large fluctuations in friction coefficient as compared to heat-treated coatings. This is attributed to the ductile nature and non-uniform structure of as-deposited coatings. Lower micro-hardness (410 ± 30 HV) of as-deposited coatings compared to hardened AISI E52100 steel ball increases the contact area between steel ball and as-deposited coating. Further, this contributes to severe plastic deformation. Higher mutual solubility between nickel and steel leads to adhesive wear; this increases the wear rate of the as-deposited coating. Low and stable friction coefficient is observed of heat-treated coatings due Hard Ni₃P phase precipitation decreased the mutual solubility between interacting surfaces leading to less adhesive wear. 'Fig. 3(b)' shows variations in friction coefficient of as-deposited and heat-treated coatings at 300°C testing temperature. It is observed that the friction coefficient is almost same for asdeposited coating and heat-treated coating (0.40). At fixed load and sliding speed, COF decreases with an increase in the operating temperature, this can be attributed to the in-situ oxides formation during the test at high temperature.

The weight loss data of as-deposited and heat-treated coatings tested under room and elevated temperature are shown in 'Fig. 4'. It can be observed that Ni-P as-deposited coating undergoes maximum wear as compared to heat treated coating at room temperature. Heat treated Ni-P coatings are approximately 3 times more wear resistant than Ni-P as deposited coating. At 300°C testing temperature weight loss of as-deposited and heat treated coatings showed minimum variation, thereby confirming severe wear rate of Ni-P coatings at high temperature. The wear rate of as-deposited and heat-treated Ni-P coatings at room and elevated testing temperatures are shown in 'Table 3'



Figure 3. Variations in friction coefficient of as-deposited and heat treated coatings at (a) room temperature (b) 300°C testing conditions against AISI E52100 steel ball



Figure 4. Bar diagram showing the weight loss of as-deposited and heat-treated Ni-P coatings at room and 300°C testing temperatures

Table 3. Wear rate of Ni-P coatings

Coating type	Wear rate (mm ³ /N-m)		
	At 25±2°C	At 300°C	
As-deposited coating	1.68E-04	2.51E-04	
Heat-treated coating	5.61E-05	2.13E-04	

4. Conclusions

The present study has investigated the mechanical and tribological characteristics of as-deposited and heat-treated Ni-P coatings at room and 300°C testing temperature. SEM analysis of the coatings revealed homogenous defect-free nodular morphology. EDS analysis revealed the presence of 7.34 wt% phosphorus content in the as-deposited coating thus confirming the deposition of medium phosphorus Ni-P coatings. Heat treatment (at 400°C) improved the surface hardness of coatings due to the formation of Ni₃P phase. Low and stable friction coefficient (~0.61) is observed for heat-treated coatings compared to the as-deposited coating when tested at room temperature. The average friction coefficient of the as-deposited and heat-treated Ni-P coatings at an elevated testing temperature (300°C) is found be minimum (~0.29). Heat-treated coating show maximum wear resistance (5.61E-05 mm³/N-m) compared to as-deposited coating (1.68E-04 mm³/N-m) at a room temperature tribological test. The wear properties of as-deposited and heat-treated coatings are degraded when tested at an elevated temperature (300°C). Thus the present study concludes that heat treatment at 400°C is not effective in improving the tribological characteristics of the Ni-P coating at elevated temperatures.

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