

Research Article

Properties Evaluation of Electroless Ni-Coated Low-Carbon Steels

**M. S. Senthil Saravanan,¹ V. Ananda,² S. P. Kumaresh Babu,² G. Ramalingam^{1,3},
A. Haiter Lenin^{1,4}, Jemal Mohammed Yimer^{1,4}, J. B. Sajin,¹ and Trijo Tharayil¹**

¹Department of Mechanical Engineering, Sree Buddha College of Engineering, Pattoor, Alappuzha, Kerala, India

²Department of Metallurgical and Materials Engineering, National Institute of Technology, Tiruchirappalli, India

³Department of Nanoscience and Technology, Science Campus, Alagappa University, Karaikudi, 630003 Tamil Nadu, India

⁴Department of Mechanical Engineering, WOLLO University, Kombolcha Institute of Technology, Kombolcha, Ethiopia Post Box No: 208

Correspondence should be addressed to Jemal Mohammed Yimer; jemalm@kiot.edu.et

Received 18 April 2022; Revised 19 May 2022; Accepted 19 July 2022; Published 8 August 2022

Academic Editor: Balasubramani Ravindran

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Electroless deposition or chemical coating is a well-established industrial technique that is gaining popularity. The purpose of the current investigation was to determine how long a heat treatment process had on the mechanical and corrosion performances of a Ni-P coating on low-carbon steel. The coating is performed on the low-carbon steel using Ni-P salt bath. The coated samples were heat treated at 400°C under atmospheric condition using muffle furnace. The heat treatment offers greater corrosion resistance and mechanical qualities. The coated samples were analysed using scanning electron microscope for morphological studies and X-ray diffraction analysis for phase change during heat treatment. According to the experimental findings, a heat treatment prior to coating with Ni-P layer increases hardness and corrosion resistance while lowering friction.

1. Introduction

An autocatalytic chemical process called electroless nickel plating (EN) is used to deposit a coating of nickel-phosphorus or nickel-boron alloy on a solid work piece made of metal or plastic. The presence of a reducing agent, in which the metal ions react to deposit metal, is required for the process [1]. It is possible to create alloys with phosphorus percentages ranging from 2 to 5 (low phosphorus) to 11 to 14 (high phosphorus) [1]. The metallurgical properties of alloys are determined by the percentage of phosphorus present. This plating technique is used to protect against corrosion and wear. By suspending powder in the bath, composite coatings can also be created using EN methods [2]. There are several advantages to electroless nickel plating over electroplating. It offers a consistent deposit independent of the shape of the work piece and is free from flux density and power supply problems. With

the right preplate catalyst, it can even deposit on nonconductive surfaces. Electroless nickel is used as a barrier coating. Rather than galvanic or sacrificial action, it protects the underlying metal by sealing it off from the environment. Due to its passivity and amorphousness, electroless nickel has excellent corrosion resistance, which, in most situations, outperforms pure nickel or chromium alloys [3]. The coating is nearly impervious to alkalis, salt solutions and brines, chemical and petroleum conditions, and all kinds of hydrocarbons and solvents when it is correctly applied. Additionally resistant to reducing acids, organic acids, and ammonia solutions is the electroless deposit. They are only significantly affected by extremely oxidising environments, like strong nitric acid. The most important maintenance requirement for industry is the replacement and prevention of corrosion in industrial components. The cost of corrosion in the United States is estimated to be 70 billion dollars per year, or 4.2 percent of the gross national product. In India, the

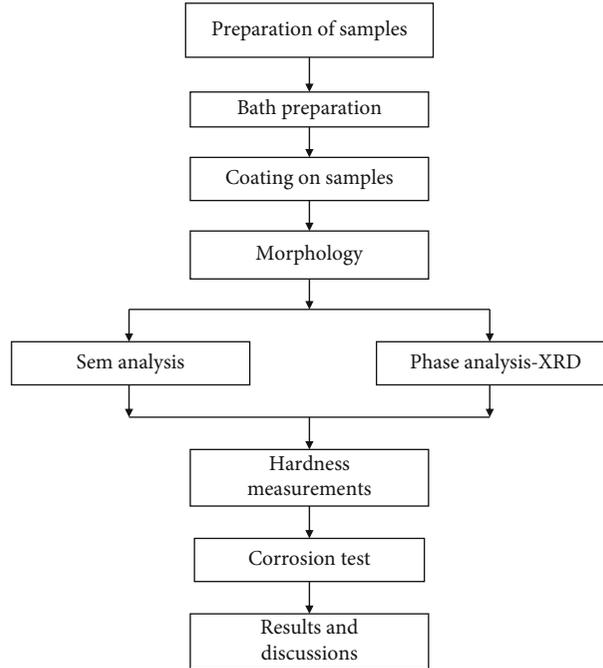


FIGURE 1: Flowchart for the experimental procedure.

annual GDP growth rate is only 4.5 percent. There are numerous methods and solutions available to prevent corrosion. To reduce corrosion, one method is to coat the metals with thin layers of less reactive metals or alloys. One of the processes that reduces corrosion is electroless nickel coating. Other, more expensive processes exist in addition to electroless nickel coating. This process is the least expensive when compared to others. Electroless deposition can be performed not only on pure metals but also on ferrous and nonferrous metals to improve surface properties. Electroless nickel coatings are well known for their unique set of properties [2]. In this paper, a cheap and economical coating technique called electroless nickel coating (EN) is used to coat low-carbon steel with Ni in this work. The coated samples are heat treated to change their state from amorphous to crystalline. The samples' corrosion resistance was compared to the impact of coating and heat treatment.

2. Experimental Procedure

The current experiment was carried out using a low-carbon steel sample. For the current work, a low-carbon steel cylindrical rod (diameter 1 cm) cut into small pieces 5 mm thick was used as the working substrate. The specimens were ground to remove corrosion and polished to achieve a smooth surface. Emery sheets were used to polish the surface to eliminate the scratches, which was then reduced in acetone, cleaned with distilled water, and dried in the absence of moisture. The specimens were then ultrasonically cleaned in deionized water. Prior to plating, all specimens were etched for three minutes in a sulfuric acid solution and then cleaned with deionized water and acetone before being weighed. Electroless nickel coating bath contains 20 g nickel

chloride, 24 g sodium hypophosphite, 40 g sodium citrate, and 25 g ammonium chloride per litre. The bath was maintained at a pH of 8 and a temperature of 363 degrees Celsius. The samples were washed in KOH at 333 K for 15 minutes prior to coating and then rinsed in distilled water. They were then rinsed one more in distilled water after spending a minute in an HCl bath. The deposition time was 2 hours, and a 70 percent KOH solution was intermittently supplied to keep the pH stable and make up for the water loss brought on by the baths [4]. The samples are classified as base metal, coated, and heat treated in a muffle furnace at 400 degrees Celsius for one hour. The heat treatment temperature is chosen based on the literature survey [5, 6]. At 400°C, the hardness of the sample gets peak value due to the transition of amorphous phase into crystalline phase of the Ni-P coating and the formation of Ni₃P precipitate which further improves the corrosion resistance. Following the coating and heat treatment, XRD is used to investigate the composition of coated samples and phase changes [7–9]. The hardness of the specimens was determined using a Vickers hardness tester with a load of 100 g and a time of 10 seconds. Finally, the electrochemical corrosion test was performed using an electrochemical corrosion tester equipped with a polarisation cell that includes an electrolyte solution (3.5 percent NaCl solution), to gauge the rate of corrosion of the samples, and a specimen holder was linked to a reference electrode, counter electrode(s), and the relevant metal sample [10, 11]. The flowchart in Figure 1 explains the details of the experiments.

3. Results and Discussion

3.1. Morphology. Figure 2 shows the SEM surface morphology of the low-carbon steel substrate, coated sample, and

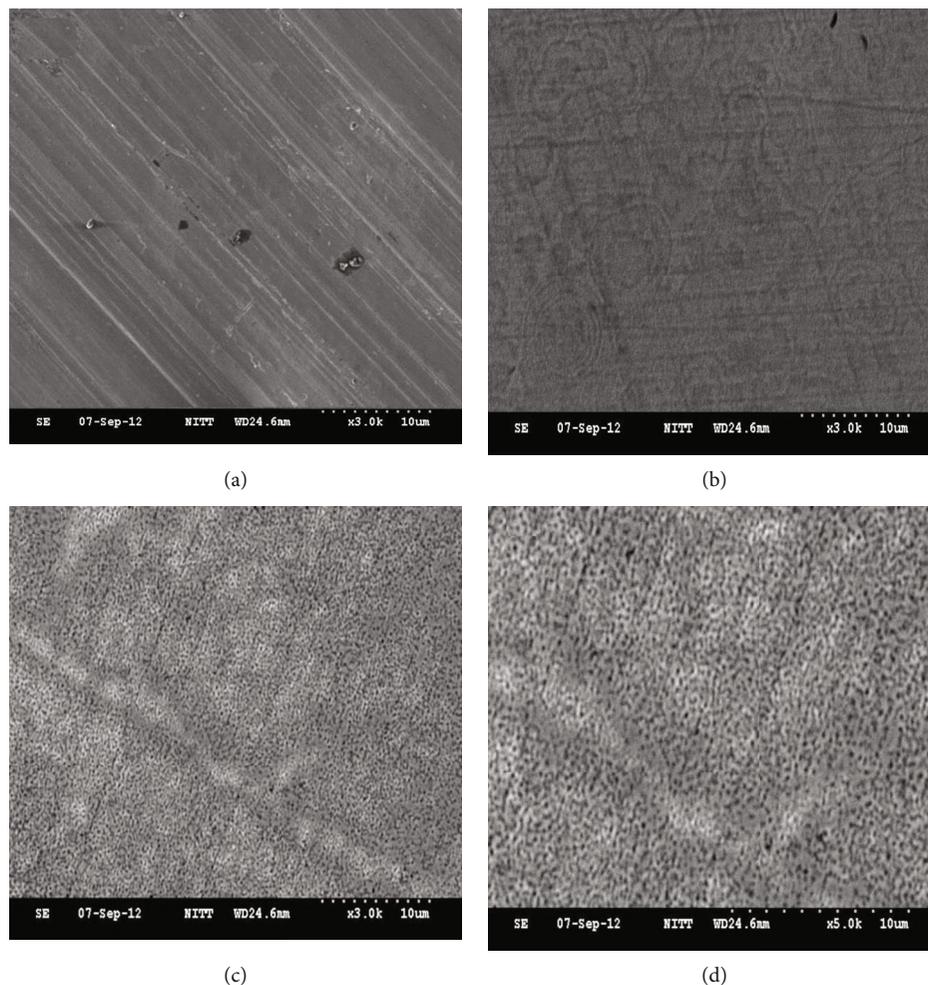


FIGURE 2: (a) Base metal, (b) coated sample, and (c and d) heat treated about 400°C/1 hour.

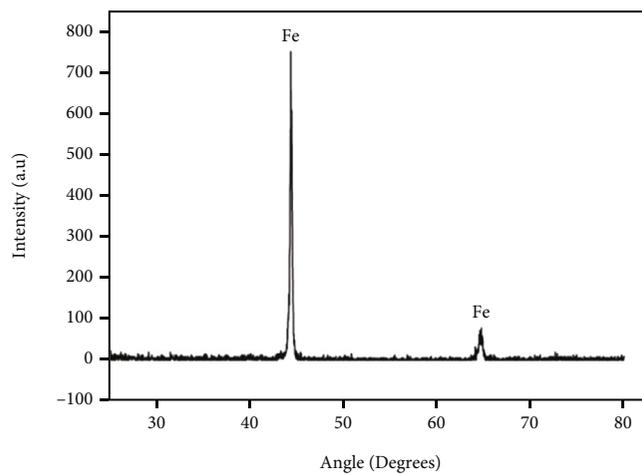
heat-treated sample. The base sample or low-carbon steel shows a clear surface, and minor particles were seen. The surface roughness of the coated sample and heat-treated samples is increased. The electroless Ni-P layers have a characteristic spherical nodular shape and are compact and homogeneous [12]. On the coated surface, there are no evident imperfections or holes, but the heat treatment changes their size and distribution. The formation of phosphides can be viewed in Figures 2(c) and 2(d) which is a heat-treated sample. Due to cracks developing in the coating as a result of heat treatment, it also affects how resistant the coating is to corrosion. The base substrate that does not undergo heat treatment is the smoothest, and remaining are rough ones. The surface roughness of the sample consistently increases which is evident in the SEM morphology.

3.2. XRD Analysis. Figure 3 shows the XRD analysis of base metal, as-deposited, and heat-treated samples. Figure 3(a) depicts the XRD patterns of a base metal containing Fe phases. Figure 3(b) shows the changes in phase after coating the sample. Because of the phosphorous content, the coated sample contains Ni and Ni₃P phases. Because of its amorphous structure, the coating is classified as high phosphorous. The crystal-

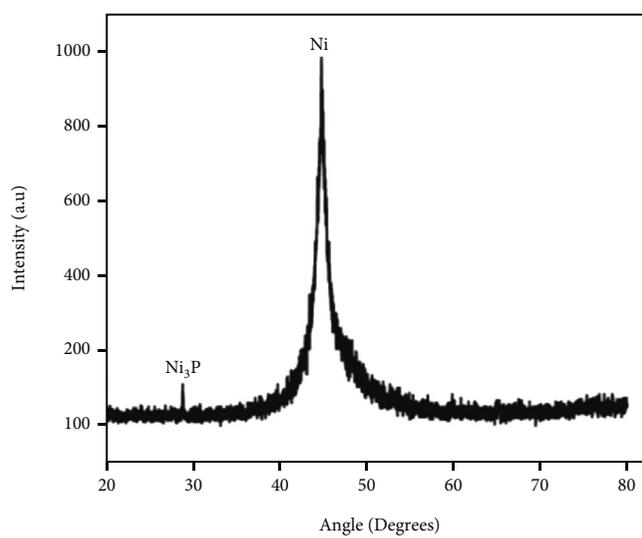
line structure of Ni-P alloy became amorphous as the phosphorus content in the metal matrix increased. The wide peak seen in the AD coating's XRD pattern at 44.5° is the same as the nickel FCC (111) diffraction peak. Due to the fine crystallite size and/or microstrain present in the coatings, XRD peak widening may be attributed [13, 14].

In Figures 3(c) and 3(d), the XRD patterns of heat-treated samples are displayed. The heat-treated samples have two peaks, one each for Ni and Ni₃P. Higher intensity and narrower peak widths suggest that the heat-treated coatings on low-carbon steel have higher levels of crystallinity and larger crystallite sizes. During the heat treatment process, all samples pass through an intermediate metastable phase [15].

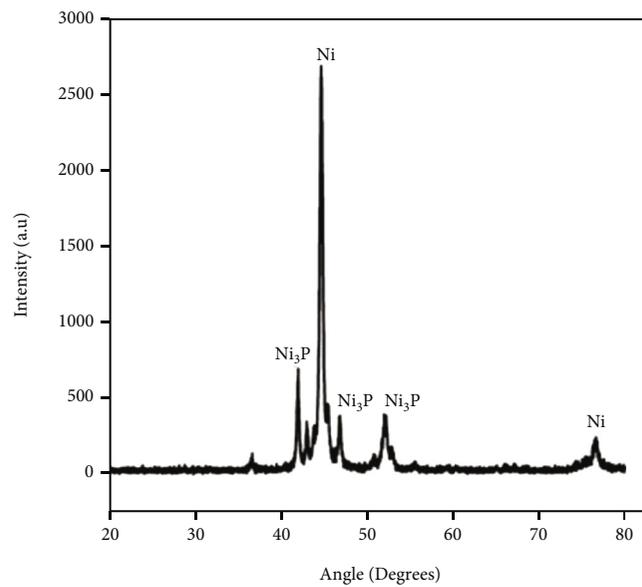
3.3. Hardness. With a 10 gf applied for 10 seconds, the hardness of the coatings and interface was assessed using a micro-Vickers hardness tester. The base metals shows a hardness of 214 HV, whereas the coated samples and heat-treated samples show high hardness values in Table 1. The increment in the hardness of the coated sample is attributed to the presence of phosphorous. The heat-treated samples shows more improved hardness value. During heat treatment, the coatings become crystalline due to the



(a)



(b)



(c)

FIGURE 3: Continued.

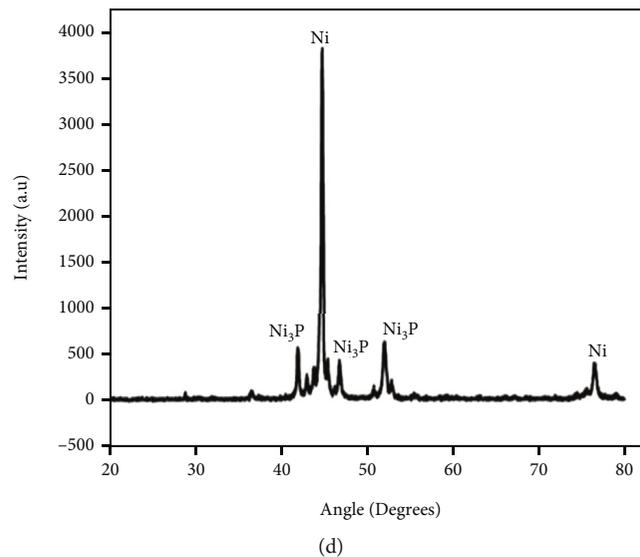


FIGURE 3: (a) XRD patterns of base metal (b), coated sample, and (c and d) heat-treated samples.

TABLE 1: Hardness values of the samples.

Sl no.	Samples	Hardness values (HV _{0.1})
1	Base metal	214 ± 10
2	Coated sample	482 ± 10
3	Heat-treated sample 1	936 ± 10
4	Heat-treated sample 2	948 ± 10

TABLE 2: Corrosion results.

Si no.	Samples	E_{corr} (mV)	I_{corr} (mA/cm ²)	Corrosion rate (mpy)
1	Base metal	630.84	0.080372	0.03
2	Coated sample	52	0.0003984	0.00019
3	Heat-treated sample 1	300.94	0.0040372	0.0019
4	Heat-treated sample 2	300.94	0.0040372	0.0019

arrangement of rearrangement of atoms [16, 17]. Heat treatment leads to the formation of hard body-centered tetragonal Ni₃P precipitate which attributed to the high hardness value. The formation of the Ni₃P precipitates was confirmed in XRD results [18].

3.4. Corrosion Results. The corrosion results of all specimens are shown in Table 2. The corrosion rate for the all the specimens was calculated by the formula below.

$$\text{Corrosion rate} = \frac{0.13EWI_{corr}}{d} \text{ mpy}, \quad (1)$$

where I_{corr} is the corrosion current in amps, EW is the equivalent weight of corroded sample, and d is the density of the corroded sample in g/cm³.

The results in Figure 4 show that the coated sample has very good corrosion resistance due to its amorphous nature. Phosphorus stimulates anodic and cathodic reactions during the corrosion process, which increases the anodic dissolution of nickel. It also increases the corrosion potential and decreases the corrosion current. Accelerated nickel corrosion creates the conditions for concentrating phosphorous and, as a result, the surface-level synthesis of stable intermediate molecules with the formula Ni₃P, which serve as a passive barrier film. The literature publi-

cations on Ni-P coatings claim that preferential nickel dissolution leads to phosphorus enrichment on the surface layer. When this enhanced phosphorous combines with water, a layer of adsorbed hypophosphite anions is created (H₂PO₂). The development of either soluble Ni²⁺ species or a passive nickel film is assumed to begin with the hydration of nickel, which is prevented by this layer by blocking the flow of water to the electrode surface. As a result, phosphorus enrichment on the electrode surface is what gives electroless Ni-P coatings their higher corrosion resistance. However, the corrosion resistance of heat-treated samples is reduced [3]. Because when the samples were heated, the electroless nickel began to crystallise and lose its amorphous nature. At higher temperatures, the particles congregate, forming a matrix of Ni₃P-forming active corrosion cells. As nickel phosphides particles form within the coating, the remaining material's phosphorous content drops. This decreases its passivity while increasing its corrosion. The particles also form small active/passive corrosion cells, which contribute to the deposit's demise [19, 20]. Heat treatment causes the deposit to shrink as it hardens, resulting in cracks through the coating that can expose the substrate to attack. The consequence is a reduction in the corrosion resistance of heat-treated samples. When nickel phosphide particles form in the coating,

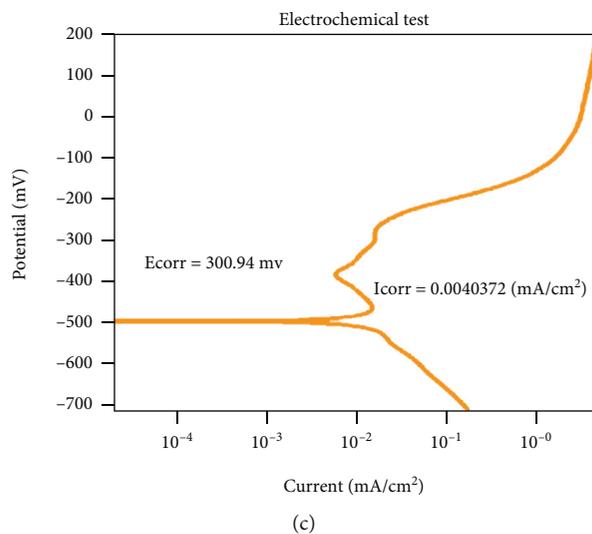
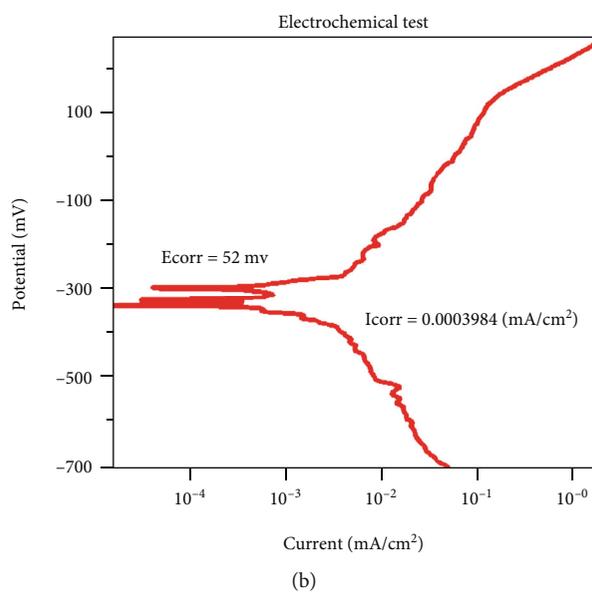
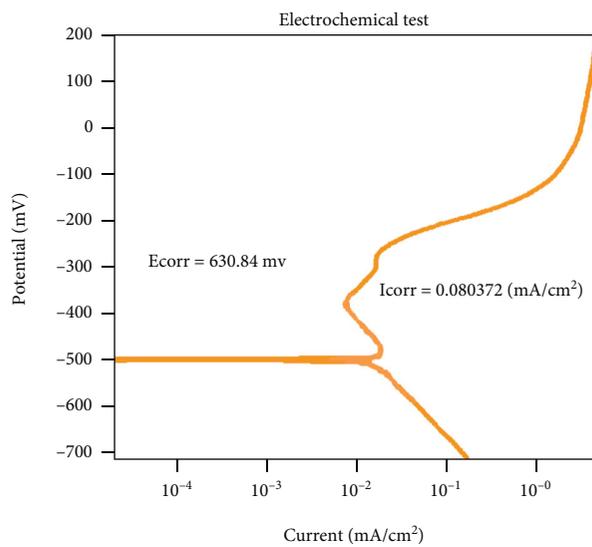


FIGURE 4: Continued.

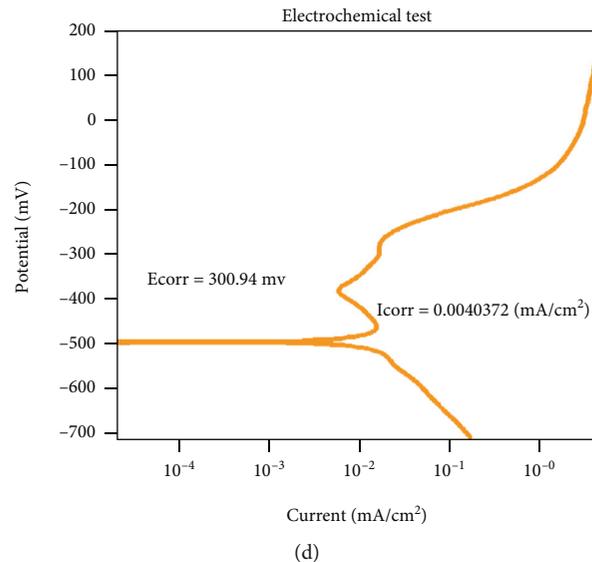


FIGURE 4: (a) Corrosion graph of base metal, (b) coated sample, and (c and d) heat-treated samples.

the phosphorous content of the remaining material decreases. The decrease in phosphorous passivity will increase the corrosion rate [21, 22].

3.5. Conclusion

- (i) Electroless deposition technique was used to successfully deposit nickel-phosphorus coatings on low-carbon steel substrates. The coated samples were then heated at 400°C for one hour
- (ii) According to the XRD examination, heat treatment of Ni-P composite coatings caused the amorphous Ni phase to crystallise and the Ni₃P phase to precipitate
- (iii) The microstructure of deposited phosphorous shows amorphous character, whereas the heat-treated samples shows high crystalline nature
- (iv) The heat-treated samples show high hardness values than the as-deposited coatings. This is due to the formation of Ni₃P precipitates

Data Availability

There are no relevant data to be made available.

Conflicts of Interest

The authors declare no conflict of interest.

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