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EVALUATION OF PHOSPHATE COATING AS A BASE FOR NICKEL ELECTRODEPOSITION ON PLAIN CARBON STEEL SUBSTRATE

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Abstract: Zinc phosphate as a pre-treatment operation was evaluated for electrodepositions of nickel on plain carbon steel substrate. Phosphate zinc bath was formulated using zinc oxide (ZnO), phosphoric acid (H₃PO₄), nitric acid (HNO₃), sodium nitrate (NaNO₃), distilled water (H₂O), and iron wool. Phosphating operations were carried out at temperature ranges of 85° C to 95° C at varied concentrations of phosphoric acid for 20 to 60 minutes. Nickel was electrodeposited on the phosphate and on as received steel substrates at 3 Volts for 30 minutes. The samples were later exposed to atmospheric air at room temperature for over 150 days. Metallurgical microscope was used to study the film adhesion and continuity as the parameters for assessing the quality of nickel electrodeposited on the phosphatized substrates. The result obtained showed that the nickel film well adhere to the phosphatized sample. It is seen that the nickel film of the sample without prior phosphate peel-off while the films of samples phosphate before nickel electroplating remained well adhered. Zinc phosphate could serve as an alternative to copper cyanide bath as pretreatment for nickel electrodeposition on plain carbon steel with improved adhesion.

Keywords: Zinc phosphate, nickel, electrodeposition, film morphology, adhesion

1. INTRODUCTION

Corrosion has been the subject of scientific study for more than 150 years [1]. It is a naturally occurring phenomenon which refers to the degradation of materials and loss of its properties as a result of reactions or interaction with its environment [2]. All categories of engineering materials are liable to corrosion attack. The severity of the attack, however, appears more pronounced in metallic materials. Reason for this is not farfetched: most metals occur naturally in combined form, and the process of metal extraction and refining involves utilization of a considerable amount of the energy value and are thermodynamically unstable until they revert to their natural (combined) form [3]. Corrosion is part of everyday life and affects, for example, industrial plants, power stations, the oil producing industry, bridges, rails and buildings. Most industrial media are usually rich in elemental gases, inorganic salts, and acidic solutions most of which influence corrosion rates and mechanisms [4-6]. Carbon steel, the most widely used engineering material, accounts for approximately 85%, of the annual steel production worldwide. It is used in large tonnages in marine applications, nuclear power and fossil fuel power plants, transportation, chemical processing, petroleum production and refining, pipelines, mining, construction and metal-processing equipment [7]. However, the concern of the engineers over the years has been the susceptibility of steels to several environmental degradations of which the major one observed has been the corrosion of steels [8-10]. In order to improve the corrosion resistance of carbon steel, they are often alloyed, or surface treated with more corrosion resistant metals such as nickel [11]. There are two main methods of depositing nickel on a metal substrate – electroplating and electroless [12]. Electroplating is the most widely used method of nickel plating, but it has a problem of poor adhesion that may be caused by poor pretreatment operations [13]. In order to procure a solution to this problem, zinc phosphate is introduced as a pretreatment operation. Phosphating is a conversion coating treatment mainly used in many industries as a surface preparation for coating by paint and increasing corrosion resistance [14-15]. A phosphate coating is produced by treating the metal with an aqueous solution containing the soluble primary phosphate and free phosphoric acid, as well as nitrate ions as accelerator to promote the oxidation and dissolution of the metallic surface, either by immersion or spray [16-17]. A wide variety of phosphate compositions is available. However, the right choice of the components and the operating conditions of the phosphate bath are made based on the nature of the material to be treated and its end use [18]. Hence satisfactory performance of surfaced components depends upon careful selection procedures, as well as good surfacing practice [19].

The use of deposited films in protective and decorative coatings is necessary for the coating of cutting tools. Thin film coatings serve as natural and artificial protective layers in many applications to reduce the effects of corrosion [20]. Oluwole et al. [21], investigated the corrosion of zinc plated low carbon steel in cocoa fluid and concluded that very thin coating (zinc) would prevent

steel corrosion in cocoa fluids for some time before corrosion occurs. Most corrosion resistant metals rely on film to provide protection against corrosion. If the oxide is tightly adherent, stable and self-healing, as on many stainless steels, then the metal will be highly resistant or immune to corrosion. If the film is loose, powdery, easily damaged and non-self repairing, such as rust on steel, then corrosion will continue unchecked.

Copper undercoat and heat-treatment have been used as a pre-treatment for nickel electrodeposition on steel, but there is still the problem of peeling, toxicity and high cost of energy [22-25]. Zinc phosphate as an undercoat offers advantages such as applicability to complex shapes, lower costs than many other coating processes, better control of deposit properties, ability to deposit very thick coating, and it is environmentally friendly [26-27]. It is accepted that the stability of phosphate coatings depends on their chemical composition and crystal structure [28-29]. These phosphate coatings are usually composed of hopeite $\text{Zn}_3(\text{PO}_4)_2 \cdot 4\text{H}_2\text{O}$ and phosphophyllite $\text{Zn}_2\text{Fe}(\text{PO}_4)_2 \cdot 4\text{H}_2\text{O}$. Hopeite has a higher alkaline solubility than phosphophyllite by Simescu and Idrissi [30]. Zinc phosphate bath was formulated which was used as an undercoat for nickel electrodeposition on carbon steel, and the results proved that the adhesion of nickel was better compared with as-received substrate.

2. METHODOLOGY

≡ Materials preparation

The medium carbon steel used for this research was sourced from Temade Nigeria Limited, Iju Ishaga, Lagos. The medium carbon steel substrate procured was 14 mm diameter and was cut into fifteen samples of length 40 mm on a lathe machine. The sectioned samples were grinded and polished on a polishing machine using emery paper of grades 60 - 800 micron grits, polishing cloth with 3 micron diamond paste in preparation for Zinc phosphate operation. Spark test was carried out on a sample to determine the chemical composition, and the result is presented in Table 1. The upper portion of the samples was drilled on the drilling machine for suspension of the sample in the bath. Each of the samples was pickled in a 0.5M H_2SO_4 solution for 2 minutes, rinsed in distilled water and further cleaned with cellulose thinner to remove grease, rinsed in distilled water and air-dried, after which it was weighed.

≡ Preparation of Phosphating Baths

The solution was prepared by dissolving zinc oxide in 100 cm³ of hot diluted phosphoric acid until saturation and the saturated solution was filtered. The filtrate was diluted with 100 cm³ of water to obtain pH of 1.9 and 1.7 for 1.0M and 1.5M H_3PO_4 bath respectively. Iron wool of 3.5 g, 40 cm³ of nitric acid and 10 cm³ sodium nitrate were added to the solution to complete zinc phosphate bath of 250 cm³. The samples were immersed in a phosphate bath for 20 – 60 minutes at temperature ranges of 85-95^o C; thereafter it was removed, rinsed in distilled water and air-dried.

The zinc phosphate coating weight on each steel specimen was determined by weighing the specimen on a digital weighing balance before and after immersing in the phosphate solution. The difference between the two weights gives the zinc phosphate coating weight on a particular specimen and is mathematically expressed as

$$W_c = W_2 - W_1(1)$$

While the actual coating weight is expressed as

$$\text{Actual coating weight} = W_c / A \quad (2)$$

W_1 - Weight of specimen before phosphate, W_2 - Weight of specimen after phosphate, W_c - Coating weight and A – Area.

≡ Nickel electroplating

Nickel electroplating of the medium carbon steel substrate was carried out at Federal Institute of Industrial Research, Oshodi (FIIRRO). The samples were pickled in 0.5M H_2SO_4 (to activate their surfaces), rinsed in distilled water and air-dried. The dried substrates were weighed using digital chemical weighing balance, and it was recorded as the original weight. Each of the phosphatized steel substrate was hung with copper wire on the cathode rod in the nickel bath while pure nickel plates were hung on the anode rod. The rectifier attached to the bath was switched on, and the voltage on it was adjusted to obtain 3 Volts. The current was allowed to flow for the period of 30 minutes before the substrates were withdrawn from the nickel bath. After which the samples were properly dried, and the final weight of the samples was taken. The difference between the original weight of the samples before electroplating and the final weight after electroplating was calculated, and the result obtained was recorded as the weight of nickel deposited on a low carbon steel substrates. It can be expressed mathematically as

$$W_d = W_f * W_i(3)$$

where W_d - Nickel deposit, W_f - final weight, W_i - initial weight

≡ Microstructural Examination

The surface morphology of the phosphate and nickel plated samples were also observed on the metallurgical microscope (Plates 1-4). The adhesion characteristics of the zinc phosphate conversion and nickel electrodeposited on the medium carbon steel substrate were examined by polishing the transverse section of the substrate with coarse carbide paper of grades 320 -600 micron grit on

laboratory polishing machine. Thereafter, the surface and edge of each substrate was viewed under metallurgical microscope for adhesion and continuity of the zinc phosphate conversion and nickel deposits. The results obtained were presented in Plates 5-6, and the samples were exposed to atmospheric air at room temperature for over a period of one hundred and fifty days.

3. RESULTS

The chemical analysis carried out on an as received carbon steel substrate used for this research is presented in Table 1. It shows that the carbon content of the steel was above 0.3% while the other elements had less than 2% of the composition. It is an indication that the steel substrate is a plain carbon steel, not an alloy steel. The results of the data obtained from phosphate and electrodeposition of nickel on the substrate are shown in Tables 2 - 3 respectively. The microstructures obtained for the phosphate medium carbon steel substrates are presented in Plates 1 and 2. The microstructures obtained from the nickel electrodeposition on both as received and phosphate medium carbon steel substrate for assessing uniformity and adhesion are shown in Plates 3-4.

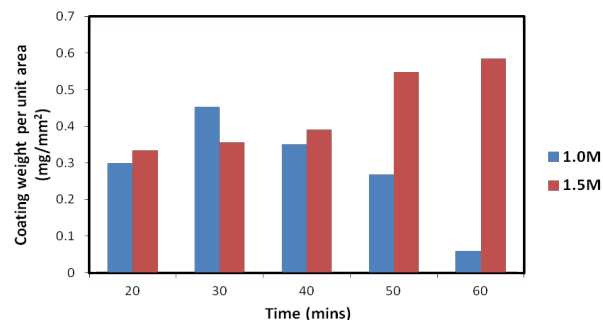


Figure 1. Coating weight per unit area of zinc phosphate on medium carbon against phosphate time for 1.0 M and 1.5 M H_3PO_4

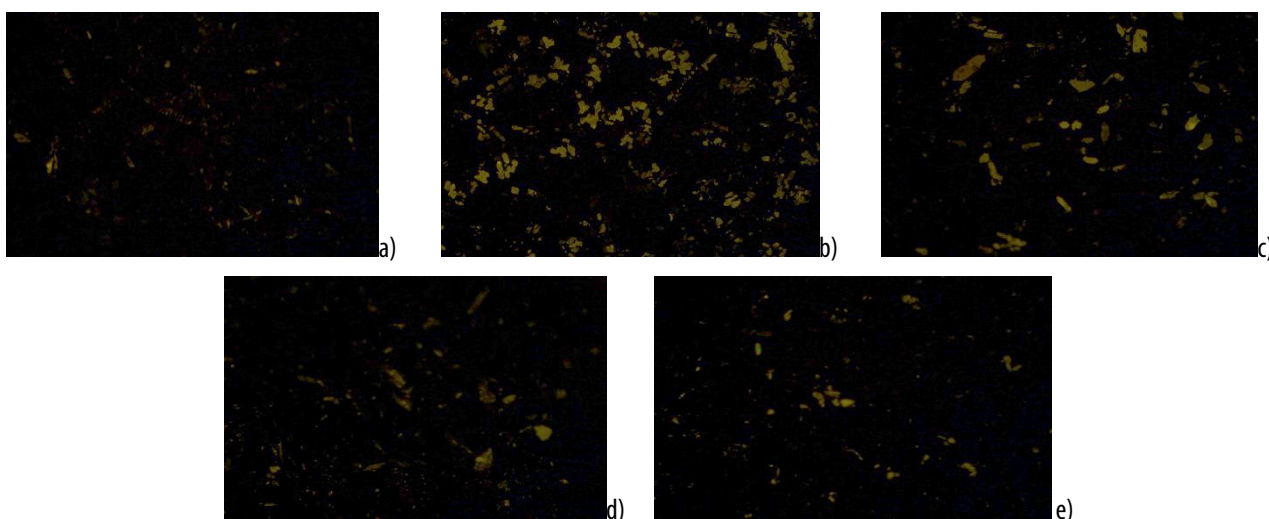


Figure 2. Medium carbon steel phosphate with 1.0 M H_3PO_4 at several minutes (a) 20 minutes (b) 30 minutes (c) 40 minutes (d) 50 minutes and (e) 60 minutes. Magnification. X 100

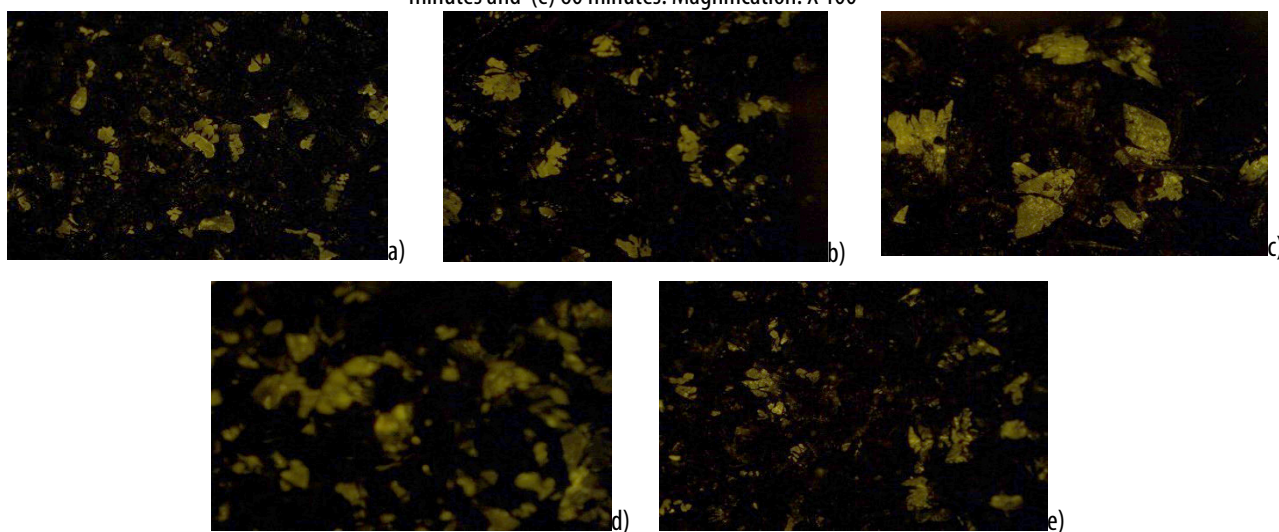


Figure 3. Medium carbon steel phosphate with 1.5 M H_3PO_4 at several minutes (a) 20 minutes (b) 30 minutes (c) 40 minutes (d) 50 minutes and (e) 60 minutes. Magnification. X 100

Table 1. Chemical composition of the medium carbon steel substrate

Chemical composition	Fe	C	Si	Mn	S	P	Ni	Cr	Other
Weight %	96.6	0.312	0.346	1.05	0.31	0.024	0.154	0.929	0.275






Sample / Observation		Photograph
a.	Light gray uniform deposit with crystalline luster	
b.	Dark gray uniform deposit with more bright crystalline luster	
c.	Dark gray uniform deposit with bright crystalline luster	
d.	Black uniform deposit with bright crystalline luster	
e.	Black uniform deposit with crystalline luster	

Figure 4. Physical observation of medium carbon steel phosphate with 1.0 M H_3PO_4 at several minutes (a) 20 minutes (b) 30 minutes (c) 40 minutes (d) 50 minutes and (e) 60 minutes






Sample / Observation		Photograph
a.	Dark gray but non uniform deposit	
b.	Dark gray but non uniform deposit	
c.	Gray, shiny but non uniform deposit	
d.	Gray, non uniform deposit and too shiny	
e.	Light gray, heavily coated and non uniform deposit	

Figure 5. Physical observation of medium carbon steel phosphate with 1.5 M H_3PO_4 at several minutes (a) 20 minutes (b) 30 minutes (c) 40 minutes (d) 50 minutes and (e) 60 minutes

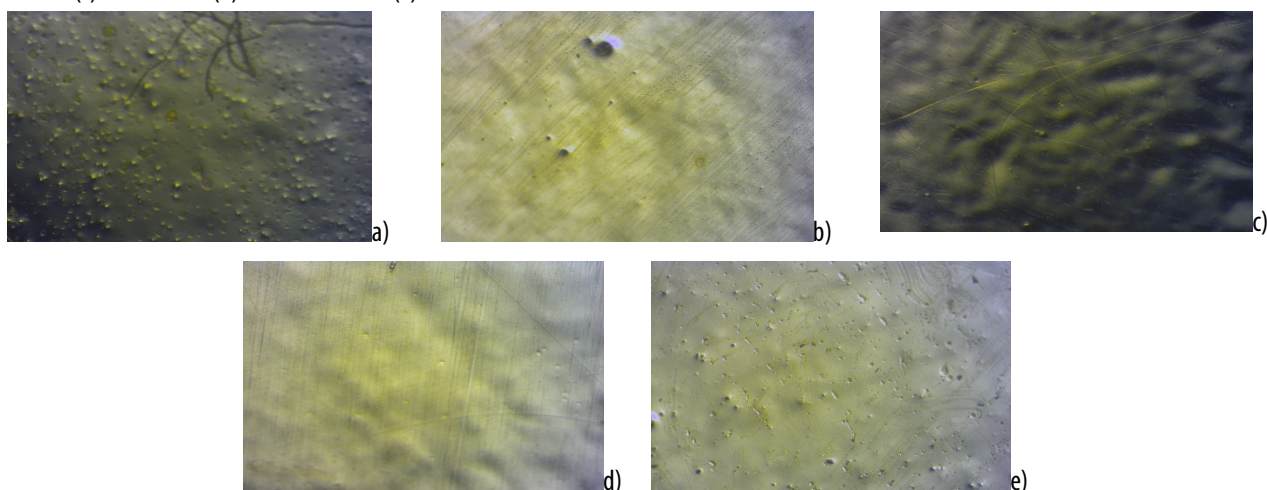


Figure 6. Micrograph of nickel electrodeposited on phosphate medium carbon steel with 1.0 M H_3PO_4 at various minutes (a) 20 minutes (b) 30 minutes (c) 40 minutes (d) 50 minutes and (e) 60 minutes. Magnification. X 100

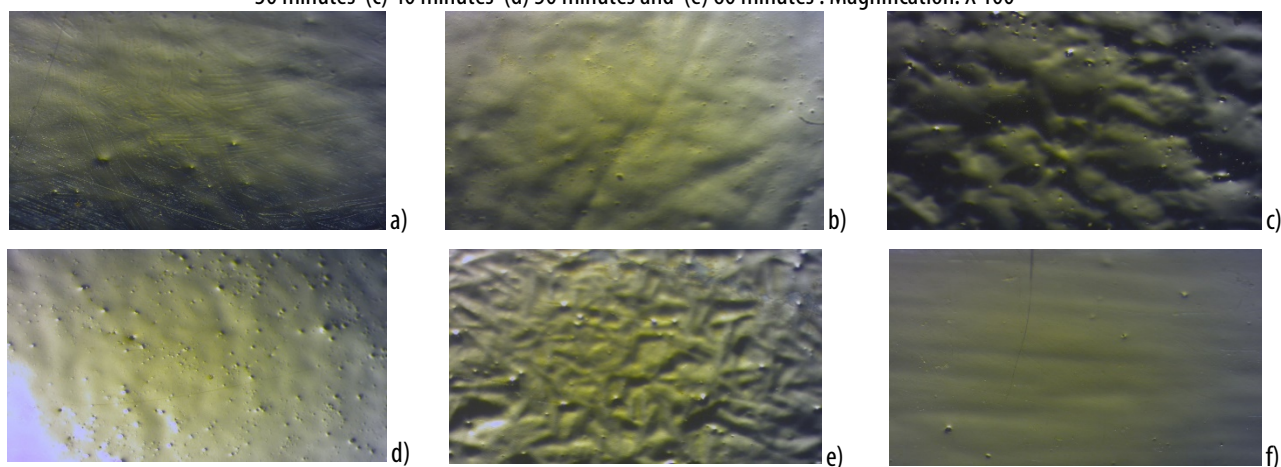


Figure 7. Micrograph of nickel electrodeposited on medium carbon steel phosphate with 1.5 M H_3PO_4 at different time and control sample (a) 20 minutes (b) 30 minutes (c) 40 minutes (d). 50 minutes, (e) 60 minutes and (f) control. Magnification: X 100

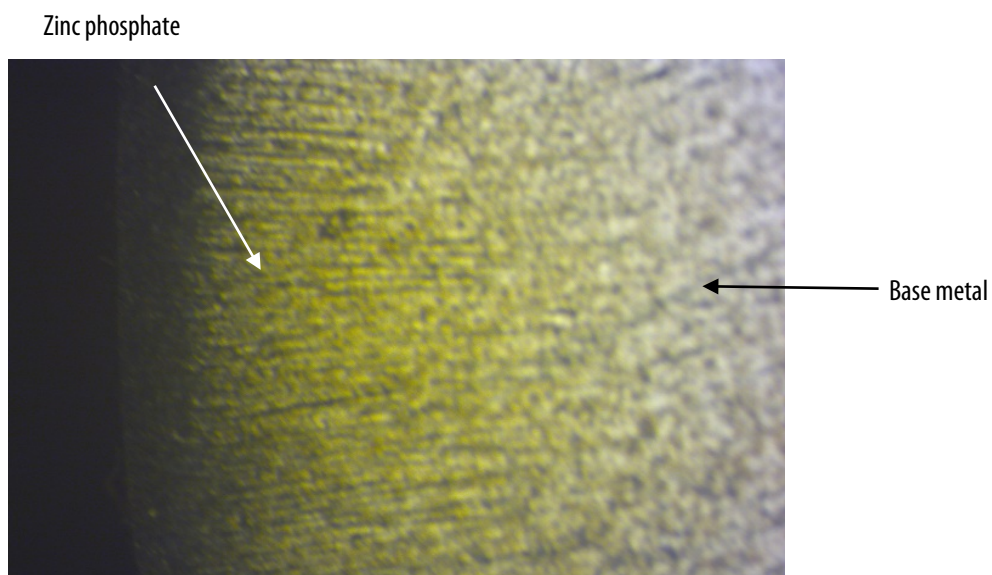


Figure 8. Cross-section of zinc phosphate conversion on medium carbon steel substrate
Magnification: X 100

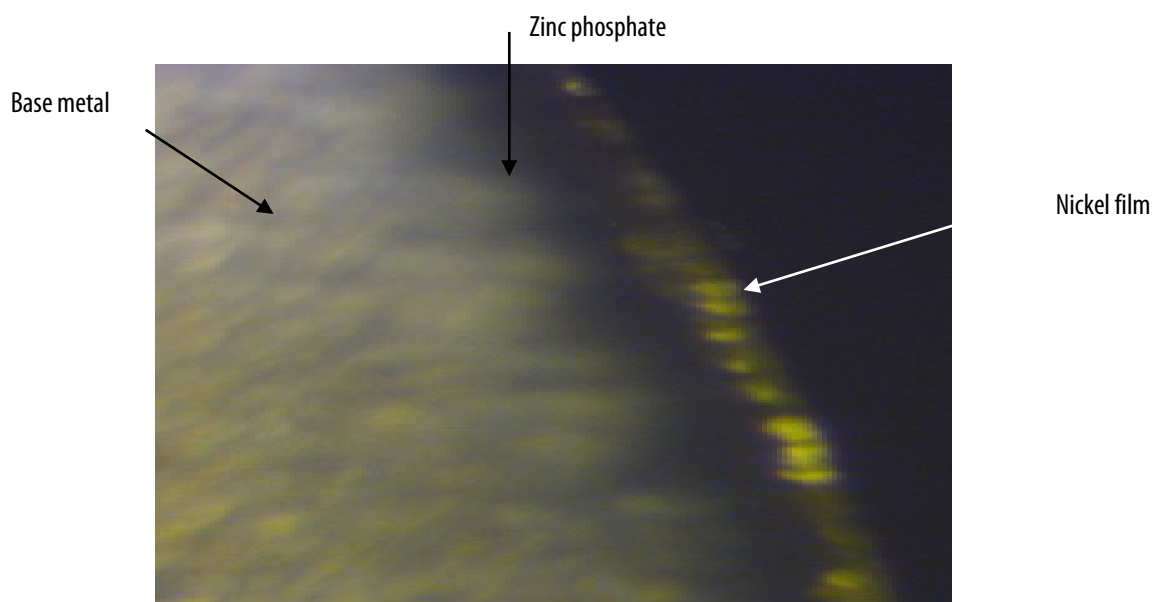


Figure 9. Cross-section of nickel electrodeposited on phosphate medium carbon steel substrate
Magnification: X 100

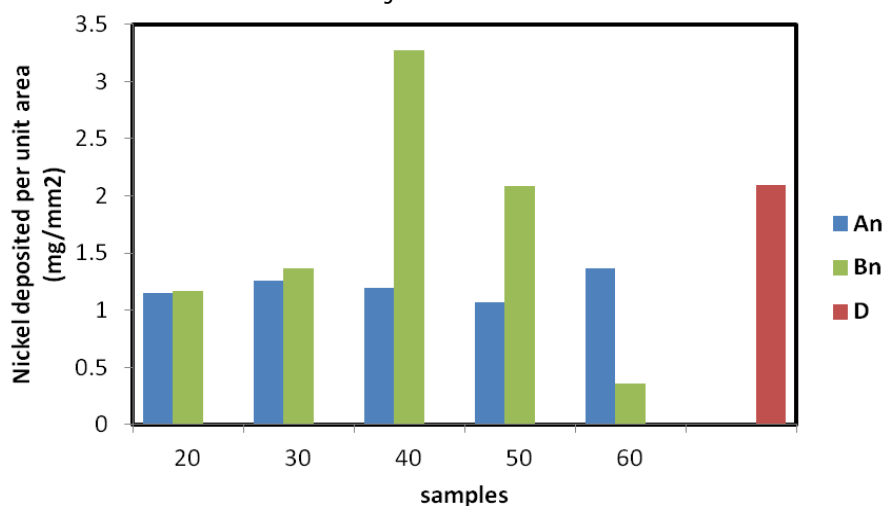


Figure 10. Coating weight per unit area of nickel electrodeposited on phosphate medium carbon steel at 1.0 M (An), 1.5 M (Bn) H_3PO_4 and control (D) steel samples at different time.

4. DISCUSSIONS

≡ Effect of concentration of H_3PO_4 on phosphate film deposition

Phosphating was carried out at 1.0 M and 1.5 M H_3PO_4 concentrations. It was seen in Figure 4 that the substrates phosphatized in 1.0 M concentration bath showed some level of uniformity in film deposition as time increases, while the substrates phosphatized in the bath containing 1.5 M concentration of H_3PO_4 (Figure 5) was not uniform and unsatisfactory with increase in time.

≡ Effect of time on phosphate coating weight

Figure 1 shows the coating weight of the substrate phosphate with 1.0M H_3PO_4 solution; it was observed that the maximum coating weight value 0.45 mg/mm^2 was obtained at 30 minutes and the minimum at 60 minutes. In other words, the coating weight increased with increase in time until 30 minutes due to the formation of phosphate grains, beyond which the coating weight starts to decrease, and this was seen to be as a result of dissolution of the phosphate grains which initially formed at 30 minutes. The colour of the coating improved dark and shiny with increase in the phosphate time as seen in Figure 4. Although all coatings are found somewhat satisfactory but the most satisfactory was that of the substrate phosphate for 60 minutes which had less lustre as compared to other substrate, uniform coating and gave the best physical appearance.

Figure 1 also presents the coating weight of substrates phosphate with 1.5M H_3PO_4 and it was seen that the coating weight of the substrate increases with time. The coating becomes thicker; the nonuniformity becomes more pronounced, and the color becomes lighter and tends towards gray with increase in the phosphate time (Fig. 5). It is seen in Figure 1, that the increase in concentration of H_3PO_4 in the phosphate bath results in an increase in the coating weight of the steel substrate. However, at 30 minutes, the coating weight at 1.5 M H_3PO_4 was slightly lower compared to 1.0 M concentration, and there was a sharp difference in the coating weight at 60 minutes. This shows that the composition of the bath (difference in H_3PO_4) affects the coating weight of the substrates at the different time [18,31]

≡ Physical appearance of phosphate coating

The colour of the phosphate coating ranged from light gray to dark gray. When strongly scratched with a fingernail, white scratch appeared on the surface but it did not cause any defect to the coating visible to the naked eye and this confirms that the coating is of good quality [18].

Figures 4 and 5 shows the photograph of all the phosphate substrates and it could be seen that the substrates coated in a bath containing 1.0 M concentration of H_3PO_4 have uniform coating and fine crystals which gave a better result than that of 1.5 M concentration which was have non-uniform coating and coarse crystals. The Figure 4 e which is characterised with finer crystal and high uniformity shows the best morphology for optimum phosphate conditions.

≡ Effect of phosphate concentration and time on surface morphology of substrate

Figures 2 and 3 displayed the micrograph of the phosphatized steel substrates at 1.0 M and 1.5 M H_3PO_4 concentration respectively. Observed on the plates are the formations of needle-like crystals that indicate that the coating is rich in phosphophyllite [32]. It was seen in Figure 2 that nucleation of phosphate grains occurred majorly in sample (a), sample (b) had a rapid nucleation and growth rate of phosphate grains and was found to decrease with an increase in the phosphate time. Figure 3(a) – (e) showed higher growth rate of crystals. From the micrographs, it was seen that increase in concentration of H_3PO_4 yielded an increase in thickness of phosphate formed and also increase on the rate of crystal formation and growth. However, substrates phosphated in the bath containing 1.0 M phosphoric acid had uniform coating while those phosphated in the 1.5 M phosphoric acid bath under the same condition were not uniformly deposited.

≡ Amount of nickel deposited on phosphatized medium carbon steel

Fig. 10 shows the chart of the amount of nickel deposited on samples phosphatized at 1.0 M and 1.5 M H_3PO_4 and control sample. It was seen in Fig. 10 that the nickel deposited on the substrate tends to increase with an increase in the phosphate time except for samples (c) and (d) from Figure 4. The decrease in the deposit on these two samples may be attributed to the surface areas of the substrates that are smaller. The quality of nickel deposited on the substrate is in agreement with the findings of Oluwole et al. [33], that coating thickness varies linearly with coating time. It is seen in Fig. 10 that, the amount of nickel deposited per unit area on the substrate (a)-(e) of Figure 5 showed an increase with an increase in time for samples (a)-(c) and decreased with increase in time for sample (d)-(e). The decrease may be as a result of the excessive phosphate coating on the substrate. The uncoated sample showed a higher nickel deposit of 2.09 mg/mm^2 in comparison with a sample with a nickel deposit on phosphatized steel substrate at 1.0 M H_3PO_4 for 60 minutes.

≡ Surface morphology of nickel films

Figure 6-7 showed the micrograph of nickel films electrodeposited on phosphatized medium carbons steel samples and a control sample; Figures 8 and 9 showed the interface between zinc phosphate and base metal and interface between the nickel electrodeposits, zinc phosphate and base metal. Brighter reflection for aesthetic value was observed on samples Figure 6(a), (d)

and Figure 7 (d). As seen in Figures 6c, 7a, b, c and e, the hill and valley shown in the nickel electrodeposition on phosphatized medium carbon steel substrate typified an uneven deposition of nickel. It is a characteristic of electrodeposition unlike electroless deposition that likely gives an uniform deposition. The roughness seen in the nickel films may be as a result of foreign particles such as air dust, turn anode bags, dropped parts, precipitates of boric acid, particles of filter carbon powder suspended in an electrolyte solution. It might also be as a result of deposition in low brightener solution at high current density. From Figures 8 and 9, the adhesion or continuous coating of zinc phosphate and nickel electrodeposition at uniformity is revealed within the limit of the power of an optical metallurgical microscope (Model AXIO Inverted) available for the research. However, nickel electroplated, phosphatized and uncoated steel substrates were exposed to atmospheric air for above 150 days, it was seen that the nickel film of the sample without prior phosphate peel-off while the films of samples phosphatized before nickel electroplating remained well adhered.

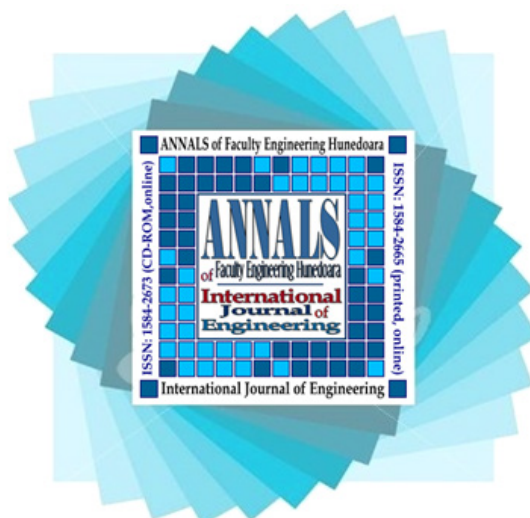
5. CONCLUSIONS

The phosphate bath formulated using 100 cm³ of 1.0 M phosphoric acid, zinc oxide, 100 cm³ distilled water, 3.5 g iron wool, 40 cm³ of 0.1 M nitric acid and 10 cm³ of 0.1M sodium nitrate had the most satisfactory and uniform coating. The films of nickel electrodeposited at 3 V for 30 minutes on phosphatized steel substrate from the formulated bath are well adhered and continuous. The nickel electroplated film on the steel substrate without prior zinc phosphate peeled off after exposure to the atmosphere at room temperature. Zinc phosphate as an undercoat for nickel electrodeposition on medium carbon steel substrate is possible and could replace the use of copper cyanide bath for pre-treatment before nickel electrodeposition.

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