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Short communication

Assessing the impact of Si_3N_4 composite films and process variable on the structural and corrosion improvement of structural steel used in construction industry

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ABSTRACT

The construction industry is faced with challenges arising from corrosion leading to structural deformation and pitting evolution of mild steel. This involved water and salt solutions forming electrolytes resulting in corrosion products. The struggle has been to provide special; desirable properties inform of coating to offer dependable service life for mild steel. This study seeks to assess the effects of deposition time of Ni-P-Zn-Si_3N_4 coatings on mild steel. The coatings were developed using direct electrolytic bath technology with constant particle concentration of silicon nitrides and different time variables. The effect of the deposition time on the substrate was assessed by electrochemical study via potentiodynamic polarization. Scanning electron microscope equipped with energy dispersive spectroscopy (SEM/EDAX) and optical microscopy was used to study the surface modification on the mild steel. It was found that deposition time affected the coating film formed and the influence of incorporating the Si_3N_4 phase into Ni-P-Zn series was seen to cause perfect nodularity and second phase crystals, especially with Ni-P-Zn-Si_3N_4 nanocrystalline deposited at 10 min. This was further established by the linear and open circuit polarization with less corroded products seen at the steel interface.

1. Introduction

One of the challenges of mild steel in the construction industry is corrosion and the consequences can be very expensive and life threatening. The causes are attributed to water / oxygen reactions combined with hydrogen at the steel cathode [1]. Another notable factor causing corrosion in construction is bacteria in the soil, which eliminates hydrogen resulting in structural destruction [2]. Corrosion problems are one area that experts are continuously working to mitigate. Since corrosion problems involve degradation and deterioration of metallic materials, research studies have shown that there can be several methods to control steel structure from excessive pit formation [3–5].

Coating technology is not only an art but a science that involves alteration of the physical, mechanical, structural, metallurgical, and chemical properties of the materials for particular applications through existing surface technology [6–8]. Research and development have established that modification of surface changes the active substrate to be modified by providing new composition, concentration, appearance, and orientation of crystals [8–10]. Powder metallurgy process offers

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Table 1
Experimental design of Ni-P-Zn-5Si_3N_4 deposition.

Sample Coating	Time (min)
Ni-P-Zn-5Si_3N_4	10
Ni-P-Zn-5Si_3N_4	15
Ni-P-Zn-5Si_3N_4	20
Ni-P-Zn-5Si_3N_4	25

opportunities for metallic materials in oxide, composite, nanocomposite forms to integrate into the different technological processes and influence the integrity and composition for special applications [9]. Among various contemporary methods is the use of electrodeposition techniques which involves electrolyte and direct electrolytic set up of direct current to provide thin film layer on structural steel [10]. Electrodeposition technology is an important technique that helps to provide unique structural modification and corrosion protection of structural steel with low cost, ease of production, compact solid structure, and thickness control [11,12]. Process parameters like time, temperature, pH, concentration of electrolyte, and stirring rate, has a profound influence on the nature of coatings produced. Also, a proper bath design can provide vivid electrochemical and structural formation for cathodic protection [13]. Inorderwords, if these factors are not properly considered, a good coating with uniform coverage may not be attained. Several reports on particle improver like TiO_2, Y_2O_3, Al_2O_3, SnO_2, Cr_2O_3, SiO_2, NiO, MgO, has demonstrated suitable ceramics and composite functional material that could provide corrosion protection in electrolyte for structural steel [14–16]. With recent interest of dual anode electrolytic deposition called electroplating process, NiZn based electrolyte has been known to render essential performance characteristics in the presence of composite and ceramics particles [1].

This study therefore seeks to assess the effects of deposition time of Ni-P-Zn-Si_3N_4 coatings on mild steel. The coatings were developed using direct electrolytic bath technology with constant particle concentration of silicon nitrides and different time variables. The effect of the deposition time on the substrate was assessed by electrochemical study via potentiodynamic polarization. Scanning electron microscope equipped with energy dispersive spectroscopy (SEM/EDAX) and optical microscopy was used to study the surface modification on the mild steel.

2. Materials and methods

Experimental design and mild steel used for this study was obtained in Nigeria and the chemical composition is shown in Table 1 and 2 respectively. All chemical used were obtained from South Africa. The metallic samples were cut into required coupons of 40 mm x 30 mm x 2 mm for steel and 60 mm x 40 mm x 10 mm with for Nickel. The samples preparation of the mild steel and Nickel surfaces follows technology process of preliminary metal cleaning [10]. $NiSO_4.6H_2O$, $ZnSO_4.7H_2O$, and $NaH_2PO_2.H_2O$ salt were obtained in powder form. The weight percentage of the $ZnSO_4.7H_2O$, is at constant concentration of 30 g/l. The bath formulation was obtained by dissolving in 1 L of deionized water 30 g of $NiSO_4.6H_2O$, 30 g of $ZnSO_4.7H_2O$, 33 g of $NaH_2PO_2.H_2O$, 60 g of $C_6H_5.Na_3O_7.2H_2O$, 25 g of $(NH4)_2SO_4$, 10 g of H_3BO_3, 10 g of Thiourea at pH of 5. The incorporated particle is 5 g of Si_3N_4. The bath was allowed to dissolve appropriately after leaving it for 24 h. The bath formulated is for quick conductivity of cation, brightener, buffers, and refiners. Requirements for electroplating are a power supply in the form of a D.C. rectifier, an electrolyte solution, a cathode, and an anode [1]. In this case, mild steel was made the cathode while the anode was 99.9 % Ni. A sulphate electrolyte with nickel and zinc-based salt was used in this study. After measurement, the compounds were put into a container. Distilled water was poured into the container, and the contents were allowed to mix and dissolve. The bath was then prepared and placed on a hot stirrer with the temperature set to 40 °C and stirring speed at 250 rpm. The coating occurred at a potential difference of 2 V, at 10, 15, 20, and 25 min. The components of the bath were changed after each time cycle in the presence of $ZnSO_4.7H_2O$. The coated samples after successful deposition were air-dried and stored in a cool and dry place. The coating characterizations were done with VEGA 3, TESCAN (SEM/EDS) and optical microscopy (OPM). The electrochemical performance was done using potentiodynamic polarization technique with an AUTO LAB PGSTAT 101 equipped with Nova 2.1. A potential difference of -1.5 V and +1.5 V and a scan rate of 0.0013 V/s was used for the polarization studies.

Table 2
Composition value of structural mild steel used.

Element	S	Mn	Si	Ni	Al	P	C	Fe
Composition (%)	0.02	0.15	0.016	0.01	0.01	0.01	0.032	Balance

Table 3
Results for Ni-P-Zn-5Si_3N_4 Coating.

Sample Coating	Time (min)	Weight Gain (grams)	Coating per Unit Area (mg/mm^2)	Coating Rate ($mg/mm^2/min$)
Ni-P-Zn-5Si_3N_4	10	0.0863	0.03596	0.003596
Ni-P-Zn-5Si_3N_4	15	0.1351	0.05629	0.005629
Ni-P-Zn-5Si_3N_4	20	0.1522	0.06342	0.006342
Ni-P-Zn-5Si_3N_4	25	0.2110	0.08792	0.008792

Table 4
Potentiodynamic Polarization table for co-deposited Ni-P-Zn-5Si_3N_4 at different time variation in 0.5 M H_2SO_4.

Sample Label	Deposition Time (min)	E_{corr} (V)	i_{corr} (A/cm^2)	Corrosion Rate (mm/y)	Polarization Resistance (Ω)
Ni-P-Zn-5Si_3N_4	10	−0.72512	0.0022231	1.2210	70.216
Ni-P-Zn-5Si_3N_4	15	−0.72612	0.0028743	1.2301	69.690
Ni-P-Zn-5Si_3N_4	20	−0.72731	0.0029213	1.2321	60.612
Ni-P-Zn-5Si_3N_4	25	−0.73121	0.0039200	1.3001	60.010

Table 5
Potentiodynamic Polarization table for co-deposited Ni-P-Zn-5Si_3N_4 at different time variation in 3.5 % NaCl.

Sample Label	Deposition Time (min)	E_{corr} (V)	i_{corr} (A/cm^2)	Corrosion Rate (mm/y)	Polarization Resistance (Ω)
Ni-P-Zn-5Si_3N_4	10	−1.10210	0.009807	1.4321	59.212
Ni-P-Zn-5Si_3N_4	15	−1.21010	0.009901	1.8543	56.234
Ni-P-Zn-5Si_3N_4	20	−1.43201	0.009976	1.8656	55.123
Ni-P-Zn-5Si_3N_4	25	−1.45211	0.012134	2.3134	53.218

3. Results and discussion

3.1. Physical and structural studies

The deposited coatings physical characteristics were accessed through weight gained and coating surface area variation, as shown in Table 3. The essential parameter of variant is the deposited time effect and the incorporated 5Si_3N_4 particle on the developed matrixes. The crystalline formation on the mild steel was seen to change as time increases due to the control of the process parameter. Ni-P-Zn-5Si_3N_4-25 min seems to have massive particle deposited but with a high agglomeration rate at the surfaces. This was seen to affect the weight gain with 0.2110 g to 0.08792 A/m^2 for weight gain and coating per unit area respectively. However, with Ni-P-Zn-10Si_3N_4-10 min, the deposition rate was lower, although with a stable mass transfer effect. With reduce time at 10 min, 0.0863 g to 0.03596 A/m^2 as weight gained and reliable surface film coverage was attained. The change in the physical characteristics of the coatings was mainly due to the different deposition time effect and nucleation of silicon nitride inclusion toward providing self-dispersing hardening behavior (Tables 4 and 5).

Figs. 1 and 2 shows the structural image of the developed deposited coating alloy on mild steel. A look at it shows that time and quantity of particle significantly affects the deposit propagation and the fundamental orientation. In Fig. 1, with 10 min coating fabrication, a good sacrificial crystal, and smaller grain were seen. Also few pores were seen all over the grain boundaries. The impact of crystal formation from the Si_3N_4 incorporation on Ni-P-Zn-5Si_3N_4 shows that there might be better grain refinement from the sulphate salt if the particle of Si_3N_4 were to increase further to close the interstitial. It is also good to establish that Si_3N_4, in its individual characteristics, affects structural build-up significantly [17]. In addition, developed coating at 25 min also provides a more circular cone-like Ni-P-Zn-5Si_3N_4 structure with visible alteration in the crystal growth due to a higher time effect. The coating at 25 min contains more significant agglomeration with non-stabilize diffusion. Even if there is an increase in time to help assist in the adsorption of an intermetallic metal grain of Silicon nitride into the intermediate of the expected coating, the stability becomes a concern due to large agglomeration seen.

The effect of nanocrystallization structure on the Ni-P-Zn-5Si_3N_4 coating is presented in Figs. 3 and 4. The linear potentiodynamic polarization and open circuit potential plots were used to inspect the electrochemical properties and surface active stability of the deposited coatings under varied time difference. From the Tafel plots, the acquired result in Fig. 3 displays the positive response and reduced corrosion rate especially for coating developed in 10 min compared to that of coating deposited at 25 min. This result is against the normal trend for possible coating where deposits of higher time

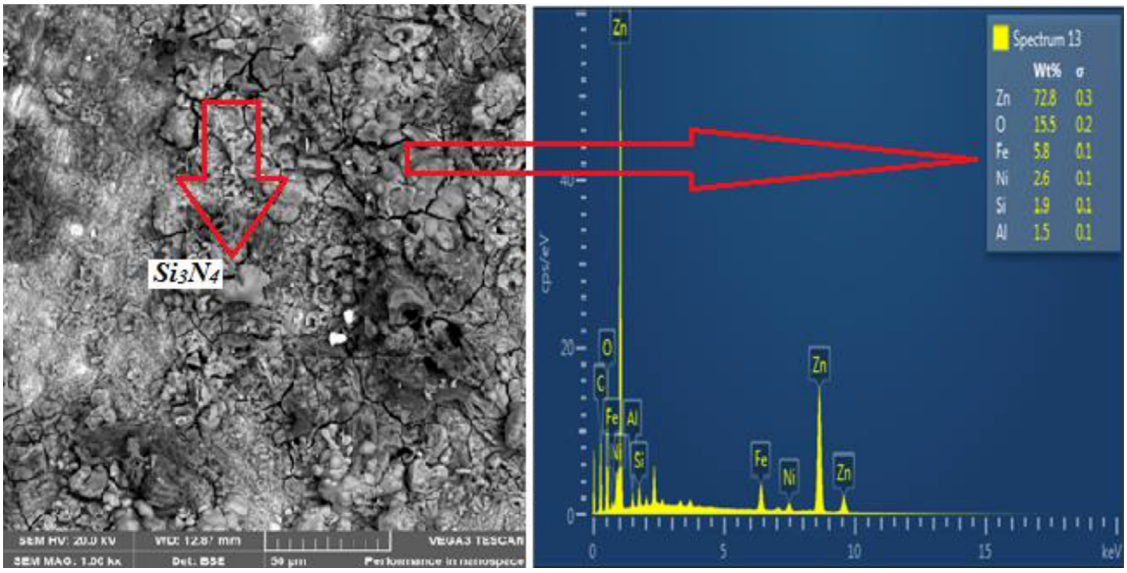

Fig. 1. SEM-EDAx crystal evolution of co-deposition of Ni-P-Zn-5Si_3N_4mild steel surface at x500 Magnification in 10 min.

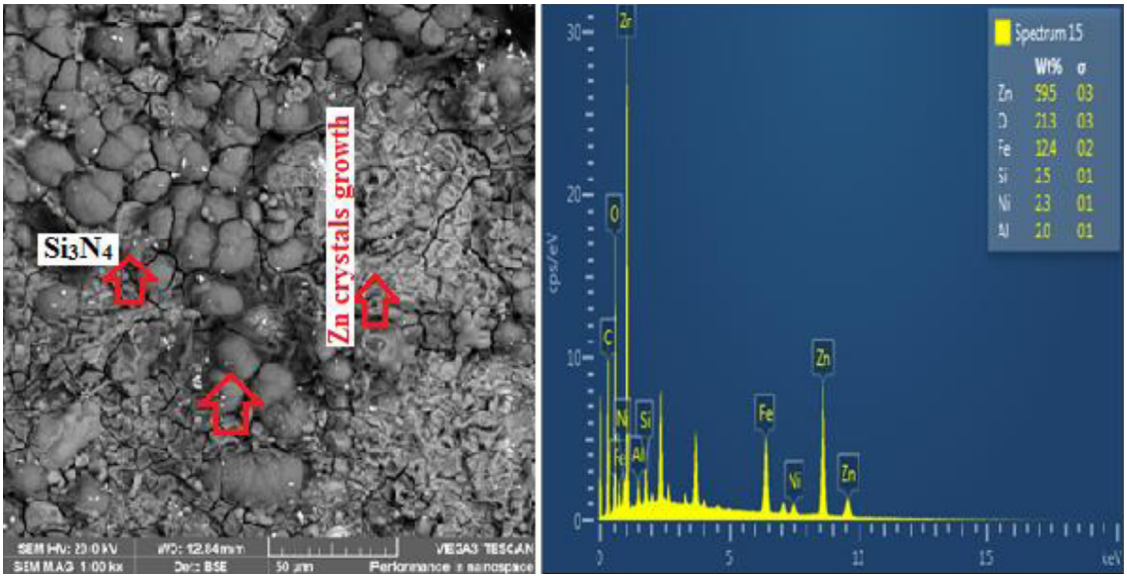

Fig. 2. SEM-EDAx crystal evolution of co-deposition of Ni-P-Zn-5Si_3N_4mild steel surface at x500 Magnification in 25 min.

function influence the nucleation behaviour and mass transport process. More so it is expected that the metal deposition rate on the cathode should be proportional to the rate of ions consumed due to the influence of surface strengthening behaviour of the mild steel substrate. On the contrary, the corrosion resistance of the developed coating from 25 min possess polarization resistance of 60.010 Ω, corrosion rate of 1.3001 mm/y and corrosion potential of -0.73121 V. Though, there is a better improved passive characteristic with coating produced in 10 min with polarization resistance of 70.216 Ω, corrosion rate of 1.2210 mm/y, and corrosion potential of -0.72512 V. It is assumed that adsorptive atom from the electrolyte during electrodeposition provides more passive effect for developed coating at 10 min than particles at 25 min with crystal forming intercrystalline within the interior of the structural steel.

Figs. 5 and 6 also demonstrated the electrochemical behaviour of all coatings made on the mild steel in 3.65 % NaCl. Since structural effect has great influence on the cathodic protection and anodic shift of the coatings in electrochemical reaction, ions of chloride was resisted with the imposed current by the Ni-P-Zn-5Si_3N_4 composite coatings. The corrosion polarization resistance for Ni-P-Zn-5Si_3N_4-10 min was 59.212 Ω, Corrosion rate of 1.2210 mm/y and E_{corr} of -0.72512 V. The Ni, P, Zn and metalloid deposited was observed to reduce in its stability with higher corrosion rate damage seen at 25 min. The corrosion

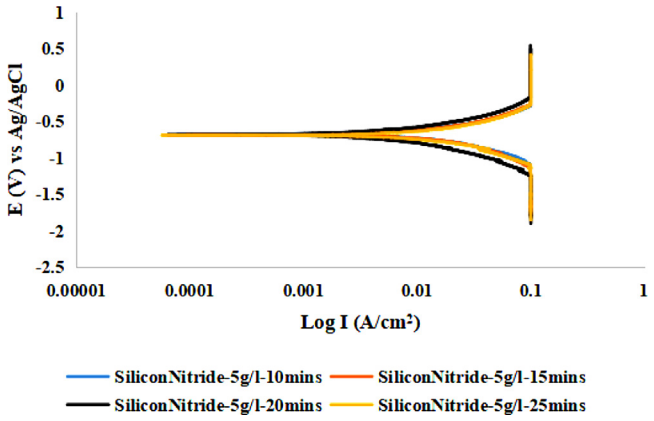

Fig. 3. Potentiodynamic Polarization curve of co-deposition of Ni-P-Zn-5Si_3N_4mild steel surface in 0.5 M H_2SO_4.

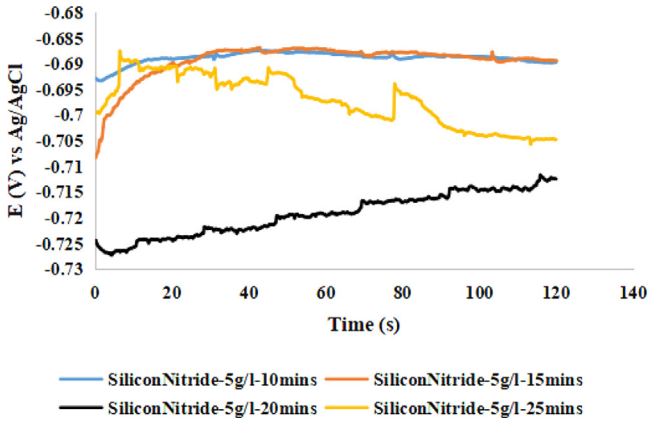

Fig. 4. OCP curve of co-deposition of Ni-P-Zn-5Si_3N_4 mild steel surface in 0.5 M H_2SO_4.

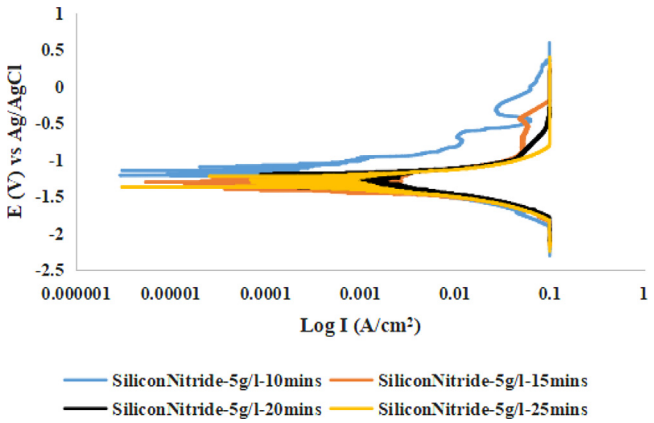

Fig. 5. Potentiodynamic polarization curve of co-deposition of Ni-P-Zn-5Si_3N_4mild steel surfacein 3.5 % NaCl.

polarization resistance for Ni-P-Zn-5Si_3N_4-25 min was 53.218 Ω, Corrosion rate of 2.3134 mm/y and E_{corr} of -1.45211 V. Although the rate of surface film deposited at the surface layer is caused by the ionic concentration from the electrolyte and time function, invariably, less coating stability at 25 min was due to poor nucleation, burnout and agglomeration. In Fig. 6, the open circuit potential also follow the same trend with potential effect observed to be -0.61 V for composite coating deposited in 10 min compared to the 1.01 V gained by Ni-P-Zn-5Si_3N_4-25 min alloy coating

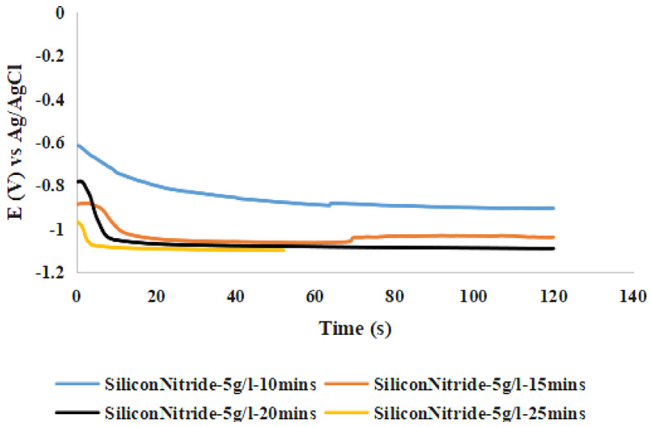

Fig. 6. OCP curve of co-deposition of Ni-P-Zn-5Si_3N_4mild steel surface in 3.5 % NaCl.

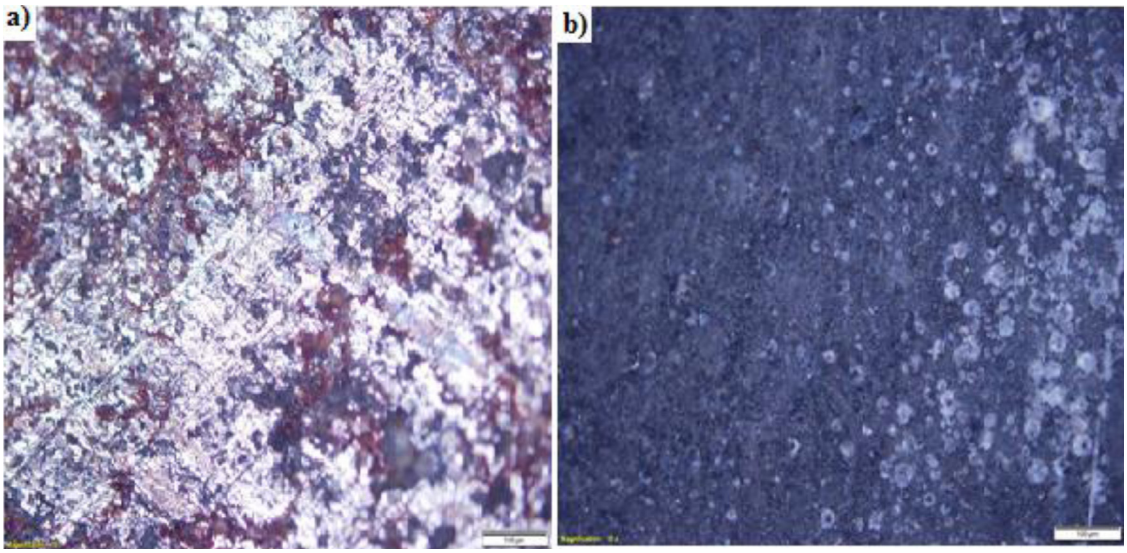

Fig. 7. OPM images at 10X magnification showing the pitting effect of corroded co-deposition of Ni-P-Zn-5Si_3N_4 mild steel surface in 3.5 % NaCl for a) 25 min b) 10 min.

The effect of deposition time on Ni-P-Zn-Si_3N_4 coating could be seen to alter the physical and structural properties of the deposited film on the substrate (mild steel). The consequences of corrosive ion on the corroded surfaces are presented in Fig. 7a and b. The optical microscopic images were taken at 10X magnification and both samples provided very clear results. The morphology of the deposited coatings deposited at 25 min showed more pitting evolution at the mild steel surfaces of coatings deposited in 25 min (Fig. 7a). In Fig. 7b, at 10 min deposit, fewer corrosion pits were seen on the steel structure and its crystal evolution even in the presence of corrosive media were retained. This is attributed to stable growth and perfect diffusion formation in response to the time of transport of ionic film species during electrodeposition. This mechanism supports the study by [18], who established that stable crystal adsorbed provides electrogrowth that blocks pores within the micro substrate, thereby providing a resilient effect against metal surface corrosion damage.

4. Conclusion

The electrodeposition route was successfully used to produce novels Ni-P-Zn-Si3N4 composite coating on a mild steel substrate at different deposition time. The different time of deposition and presence of silicon nitride nanoparticle incorporated into the sulphate bath resulted to changes in the morphological structures of the deposited films. Positive nucleation of silicon nitride propagation was seen to reduce corrosion damage on the steel structure in both sodium chloride and sulphuric acid media. The reduced microcells in the corroded surface of coating fabricated in 10 min is ascribed to the stable strengthening effect of the nanocomposite particle within the pore lattice.

Declaration of Competing Interest

The author will like to declare that this research has no financial or work related competing interest.

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