
Corrosion of Heat Treated Electroless-Ni Plated Mild Carbon Steels in Dilute H₂SO₄

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Abstract: The corrosion of heat-treated Electroless-Nickel (EN) plated mild carbon steel (MCS) in dilute H₂SO₄ is investigated and reported in the study. The MCS samples were electroless-Ni plated in alkaline Nimudeen 850. EN plated samples were soaked at different selected temperatures between 150-370 °C at constant time. The weight loss of the specimens by full immersion method was employed to assess the corrosion damage and corrosion rates of the as-received, EN plated MCS and four heat treated EN plated samples in the 0.5 M H₂SO₄ solution. The test specimens were regularly examined at every 12 hrs interval for 30 days (720 hrs). The materials corrodes in 0.5 M H₂SO₄ in the order of MCS10 > MCS14 > MCS15 > MCS11 > MCS13 > MCS12. Based on the results obtained, it will be justified to choose EN-plated samples (MCS 11) and the heat treated EN film (MCS12 and MCS13) for the purposed of solving the problem of corrosion in the medium. This is because; the application of EN deposition reduced the corrosion of MCS at much lower rate than the heat-treated EN film (MCS14 and MCS15) in 0.5 M H₂SO₄ environment. Atomic Absorption Spectrometer (AAS) Thermo series 2000 model and Scanning Electron Microscopy (SEM) with the Energy Dispersive X-Ray (EDX) facilities (Jeol JSM-7600F model) were used to ascertain the chemical compositions and the microstructures of samples.

Keywords: Corrosion, Heat Treatment, Electroless-Ni Plating, Mild Carbon Steel, Protective EN Film

1. Introduction

Several structural alloys including steels corrode simply from contact with moisture in the air, but the process can be strongly affected by interacting with certain acidic, neutral or alkaline substances. Protective film could be obtained with proper pre-treatment cycles and correct process control, good adhesion and brilliant service performance, very homogeneous thickness all over the component shape and size. Coating a surface with a thin film modifies the material surface characteristics, and it is a significant tool for controlling friction and wear at the surface [1]. Electroless nickel (EN) plating is a process of depositing a nickel alloy from aqueous solutions onto a substrate without the use of electric current. This is contrary to electroplating which relies on external source of direct current to reduce nickel ions in the conducting electrolyte to nickel metal on the substrate [2, 3, 4]. EN plating is an auto-catalytic chemical reduction

process by which nickel ions is reduced to nickel metal in solution such as in sodium hypophosphite. It is used to deposit nickel-phosphorus or nickel-boron alloy on metal or plastic substrates [5, 6]. The process is based on the presence of a reducing agent (usually hydrated sodium hypophosphite (NaPO₂H₂·H₂O)) which reacts with the metal ions to deposit metal (Ni) [7, 8]

EN is often a requisite to offer corrosion fortification and wear resistance. Heat treatment is frequently used to enhance the surface wear resistance properties [9] for their high hardness and good, natural lubrication characteristics [2, 10]. EN deposits have low ductility and phosphorus content (%) within the regular operating range has very little effect. EN deposits are hard and brittle film. The customary heat-treatment measure for EN increases the hardness and reduces ductility, and thus wear resistance can be improved by heat treatment [11].

The corrosion resistant property of EN makes it very useful as a protective coating, though all EN deposits do not

behave the same way [12, 13, 14]. High phosphorus deposits are comparatively substandard to low phosphorus deposits in high temperature and strongly alkaline media but are more efficient in neutral or acidic media. EN does not serve as a sacrificial coating in the same manner as zinc or cadmium does on steel substrates to provide protection against corrosion. It acts as a barrier coating between the corrosive media and the substrate. Thus, the film thickness and the absence of pores in the deposit are of great significance [2, 8, 15, 16].

EN has exceptional corrosion resistance and it is not vulnerable to stress corrosion cracking and can be deposited with a variety of composition for use in acidic, neutral or alkaline environments [17, 18]. It is known that the surface finish of the substrate can appreciably influence the protective value of EN deposits; while smoother substrate surfaces give better quality electroless deposits [2]. Surface deficiencies often results from mechanical surface treatments and fabrication procedures such as rolling, stamping, drawing, shearing, lapping, machining, grit blasting and casting [19, 20].

Protective EN films (heat treated) with good adhesion [21] and homogeneous thickness often comes with distinct advantages when plating valves or threaded parts, holes, recesses, irregularly shaped objects and internal surfaces [8, 22].

Due to the hazardous nature of some metal coating processes such as the cadmium plating; alternate materials have been sought for many years by the manufacturing industries around the world. With respect to this, the

corrosion of Zn-Ni, Ni-P, and Ni-B deposits and the appraisal of their prospect as an alternative to cadmium have been extensively studied using different approach [23].

Several structural alloys including steels corrode simply from contact with moisture in the air, but the process can be strongly affected by contact with certain acidic, neutral, basic or alkaline substances [24]. Numerous studies have been carried out on the corrosion of mild carbon steels in various media such as HCl, H₂SO₄, NaOH and NaCl [25]. Reports are also available on investigation of some control measures such as the application of electroplating [3,4,26]; galvanising; chemical vapour deposition (CVD) and plasma vapour deposition (PVD) [27, 28]. Hence the corrosion of heat treated EN plated MCS in dilute H₂SO₄ is investigated and reported in the present study.

2. Methodology

2.1. Materials and Sources

300 cm length of 25 mm diameter mild carbon steel (MCS) rod samples and plating chemicals were collected from the Rolling mill Section of the Ajaokuta Steel complex, Kogi state, Nigeria. The plating chemicals including *Nimudeen 850*[®], *Replenisher I* and Standard grade of H₂SO₄ were obtained from the Nigeria Machine Tools NMT, Osogbo, Nigeria. The Chemical compositions of MCS samples are presented in Table 1.

Table 1. Chemical composition of MCS samples.

Sample	%Si	%Mn	%P	%S	%Cu	%C	%Fe	C.E	UTS (N/mm ²)
MCS-10	0.182	0.747	0.0094	0.015	0.203	0.351	98.493	0.516	631



Fig. 1. The pictorial view of mild carbon steel samples used for the tests.

2.2. Preparation of Samples

The as-received mild carbon steel rod samples were cut to

1.5 cm thickness and were polished using different grades of carbide paper grits which include 60 μm, 120 μm, 220 μm, 320 μm, 400 μm and 600 μm on the grinding and polishing machines. The specimens were thoroughly cleaned and rinsed in water at room temperature for 1 minute. The initial weight *W_i* was determined using a digital weighing machine. It was thereafter immersed for 1 minute in the emulsifier containing a mixture of Kerosene and Detergent solution necessary for the removal of soils (oil and grease stains) at operating temperature between 55-65 °C. This was followed by alkaline cleaning and acid cleaning using 0.4 g/l NaOH and 0.01M HCl solutions respectively operating between 60-70 °C for 1 minute. The surface of the specimen was activated by dipping in surfactant bath made of palladium chloride PdCl₂ solution (0.04 g/l) at 85 °C for 20 seconds. The specimen is rinsed from time to time in water at room temperature.

The pre-cleaned sample was submerged in EN plating bath (of alkaline reduced EN plating solution) for 10 mins. The EN plating bath consists of ratio 1 to 4 of *Nimudeen 850*[®] to distilled water, and 3.5 g/l of *Replenisher I*[®] with 5 g/l H₂SO₄. The EN plating temperatures were varied as 80, 85 and 90 °C. The EN plated sample was removed from the EN plating tank, rinsed in water and immersed in anti-tarnish chemical

consisting 5 g/l potassium-di-chromate solution at temperature range between 50 and 65 °C for about 20 seconds. The EN plated samples were dried in oven and kept in the desiccators before the final weight W_f is determined using a digital weighing machine (model DT-502A, 0.0001 g).

The amount of EN deposit (ΔW_p) on the MCS substrate is determined from the difference between the initial weight (W_{pi}) and the final weight (W_{pf}) before and after immersion plating respectively. The difference is mathematically expressed as in (1):

$$\Delta W_p = W_{pf} - W_{pi} \quad (1)$$

where W_{pi} = initial weight of the sample before EN plating.

W_{pf} = final weight of the sample after EN plating.

The amount deposited per unit area was calculated from the amount deposited as the ratio of electroless-nickel film weight deposited to the total surface area of plated sample [3].

2.3. Heat Treatment of EN Plated Mild Carbon Steel Samples

EN plated samples (MCS12, MCS13, MCS14 and MCS15) were placed in the heat treatment furnace and fired at different selected soaking temperatures and constant time as presented in Table 2.

Table 2. Heat treatment of EN plated mild carbon steel samples.

Sample No	Treatment Description	Soaking Temp. (°C)	Soaking time (min)
MCCS10	Not plated, Not heat treated	-	-
MCS11	Plated, Not heat treated	-	-
MCS12	Plated, Heat treated	150	60
MCS13	Plated, Heat treated	210	60
MCS14	Plated, Heat treated	250	60
MCS15	Plated, Heat treated	370	60

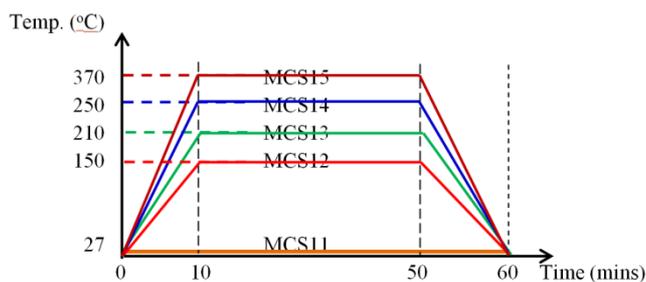


Fig. 2. Heat treatment cycle used for the experiment.

2.4. Determination of Corrosion Rates of Heat Treated EN Plated MCS Samples in 0.5 M H₂SO₄

The weight loss of specimen by full immersion method was employed to assess the corrosion damage and corrosion rates of the as-received and EN plated MCS samples in the 0.5 M H₂SO₄ solution. The initial weights (M_1) of the samples were measured immediately after the surface cleaning using an electric digital weighing machine measure to 0.0001 g. Samples were fully immersed in plastic vessels containing 25 cm³ of the test media (0.5 M H₂SO₄).

After each immersion time, the specimens were removed, rinsed in water and dried with cotton wool to remove water and other impurities before the final weights (M_2) were determined. The chemical corrosion weight loss was calculated from the difference between the initial weight (M_1) and initial weight (M_2) of the specimen before and after immersion respectively. The as-received and EN plated MCS specimens were regularly examined at every 12 hrs for 30 days (720 hrs). The same experimental procedure was carried out repeatedly on the EN plated the MCS samples.

The corrosion rate was calculated in mils per year using Equation 2 according to ASTM as reported by Fontana [29], Oloruntoba [30]; and Ajibola and Oloruntoba [3].

$$\text{Corrosion Rate (R)} = (K\Delta W_c) / (\rho A t) \quad (2)$$

where Weight loss to corrosion ΔW_c (g) = $W_{ci} - W_{cf} = (M_1 - M_2)$ (3)

W_{ci} = Initial weight (M_1) of the sample before corrosion test.

W_{cf} = Final weight (M_2) of the sample after corrosion test.

ρ = Density of the material

A = Total area of exposure

t = Exposure time

K = 534 (Corrosion rate constant)

The corrosion impact on the appearance, colour and roughness of substrate surfaces were also examined.

2.5. Metallurgical Studies on MCS Substrates and EN Plated Samples

The chemical characterisation and micrography were used to understudy the metallurgical properties such as chemical composition, roughness and microstructures of the substrate specimens (as-received and EN plated MCS samples). The chemical analyses were performed using Atomic Absorption Spectrometer (AAS) Thermo series 2000 model. The physical quality such as the brightness, appearance, colour and roughness of EN depositions were studied using high resolution microscopic camera (Samsung ST65/HD5X-14.2 model). The EN film deposition was characterised by Scanning Electron Microscopy and Energy Dispersive X-Ray analyses using Jeol JSM-7600F Field Emission SEM/EDX. The SEM photo-micrographs and EDX spectra lines data produced were used to discuss the results.

3. Results and Discussion

3.1. Determination of Electroless-Nickel Plating Parameters on Steel Substrates

The effect of the heat treatment on the morphologies (appearance, adhesion and micro-structures) and corrosion of EN deposition on MCS substrates were examined. Figs. 3-5 compare the different surfaces of the specimens. The Fig. 3a

shows the appearances of the as-received sample (MCS10) prior to its chemical cleaning and (b) the EN plated but not heat-treated sample (MCS11) which is characterised with uniform layer of the rich electroless-nickel film.

The effects of heat-treatment on the surface appearances (lustre) of EN plated MCS(12-15) samples baked at 150-370 °C soaking temperatures are shown in Fig. 4(a-d). The chemical characterisation as revealed by EDX spectral data (Table 3,) shows that the targeted area of the SEM in Fig. 5a consists of very rich nickel-phosphorus film, laid over the chromate activated MCS sample. The corresponding EDX

data (Table 3) reports about 60.34 % Ni and 10.57 % P for the 10 mins of EN plating on chromate activated MCS substrate. Under similar SEM/EDX observation; the black portions of the Ni-P rich EN deposition shows that the 100 um size SEM electron image (Fig. 5b and Table 4) of EN plating contains 11.94 % P, and 51.78 % Ni with traces of 0.52 %Zn and substantial amounts of 1.11 % Pd, being deposited from the zincate and PdCl₂ activated solutions respectively (Table 4). This shows that the EN film is more of the Ni-P film than the metallic Ni deposition than as it is obtainable in the electroplating process [30].

Table 3. EDX Spectrum processing data for 10 mins of EN plating on chromate activated MCS sample.

Element	C	O	Na	Fe	Si	Mn	Cu	P	Cr	Ni	Total
Weight %	3.36	4.35	2.11	7.34	3.67	3.69	2.26	10.57	1.31	60.34	100

Table 4. EDX Spectrum processing data of Ni-P rich EN deposition on MCS substrate.

Element	C	O	Na	Si	P	Fe	Cl	Ni	Mn	Cu	Zn	Pd	Total
Wt %	7.21	6.88	0.85	6.11	11.94	6.24	0.91	51.78	3.51	2.85	0.52	1.11	100

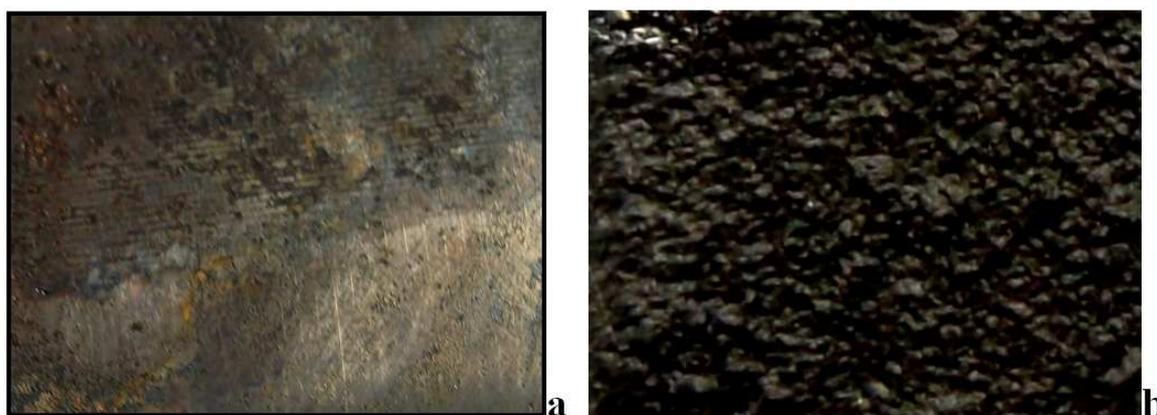


Fig. 3. Showing (a) As-received sample (MCS10) and (b) EN plated, not heat-treated sample (MCS11) (x50 mag.).

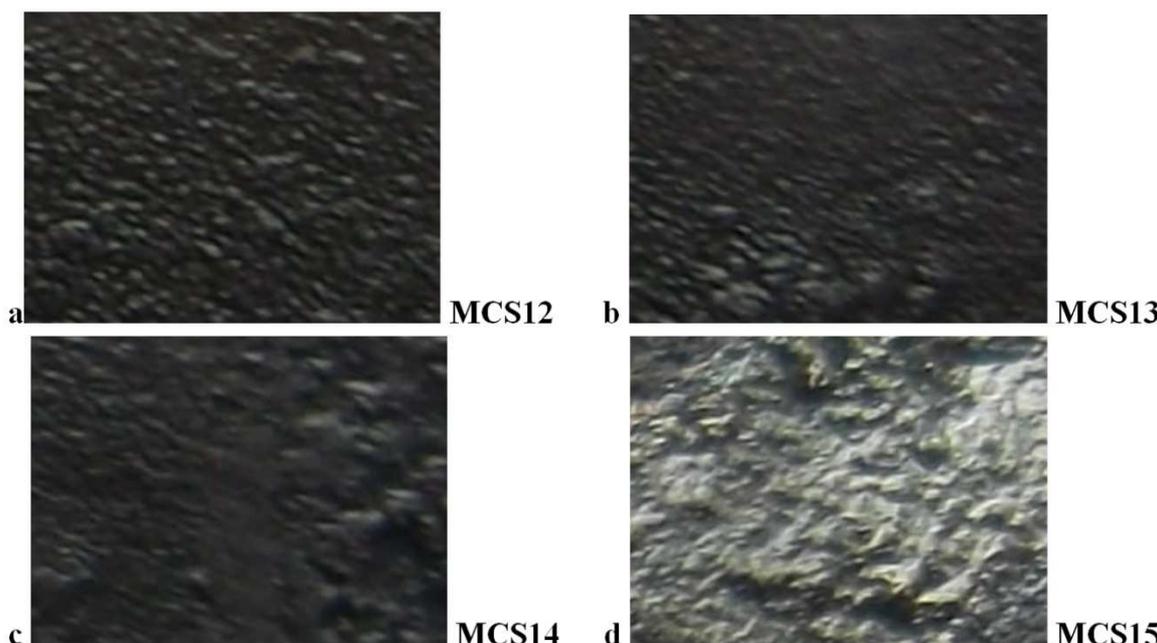


Fig. 4. EN plated, heat-treated MCS samples at (a) 150, (b) 210, (c) 250 and (d) 370 °C soaking temperatures (x50 mag.).

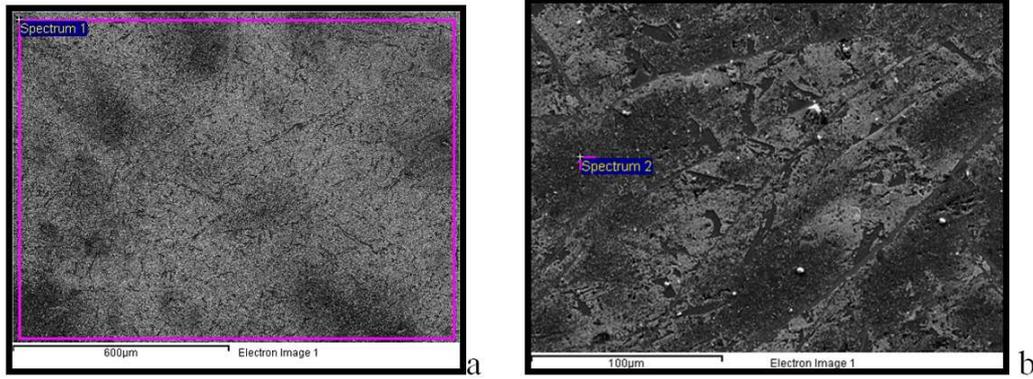


Fig. 5. SEM micrographs of (a) EN plating (x500 mag.) and (b) Ni-P rich portion on MCS (x1000 mag.).

3.2. Effects of Heat Treatment on EN Film

Figs. 6(a,b), 7 and Table 5 compare the SEM electron images of EN plating on MCS 11 (Fig 6 a,b) and the heat treated EN plating on MCS15 (Fig. 7). The observed portion of the heat treated EN deposition shows that the 100 um size

of EN plating contains 10.39 % P and 61.07% Ni as revealed in the EDX chemical characterisation data. The EDX data possibly shows that there was a little reduction in the %P resulting from the hardening treatment held at 370 °C furnace temperature.

Table 5. EDX Spectrum processing data of EN plated chromate activated steel substrates (MCS11, MCS15)

Element (Wt %)	C	O	Na	Fe	Si	Mn	Cu	P	Cr	Ni	Total
MCS 11	6.38	7.05	1.47	5.31	2.62	3.01	2.04	10.37	0.35	61.40	100
MCS 15	5.73	7.43	1.45	8.20	1.34	2.30	2.19	10.29	1.21	61.07	100

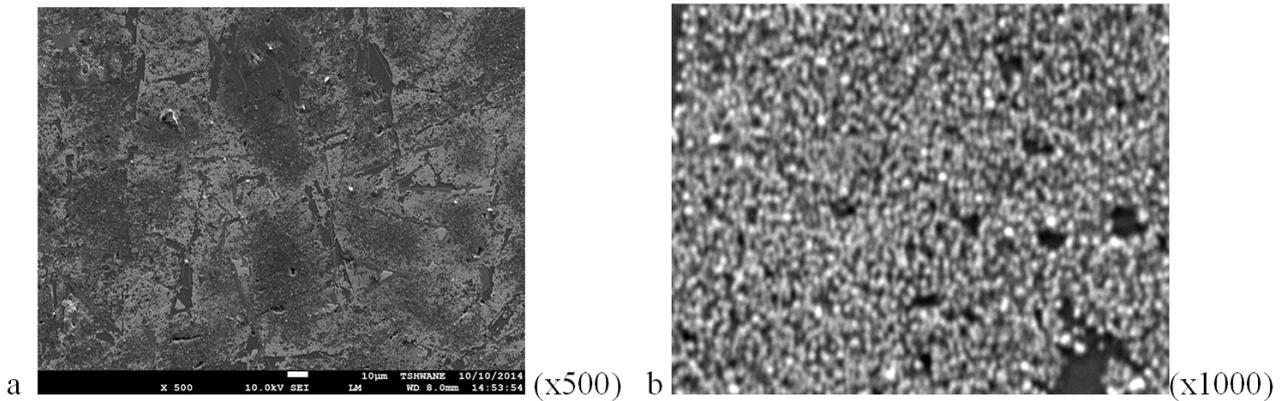


Fig. 6. SEM showing surfaces (a,b) of EN plated mild carbon steel (MCS11).

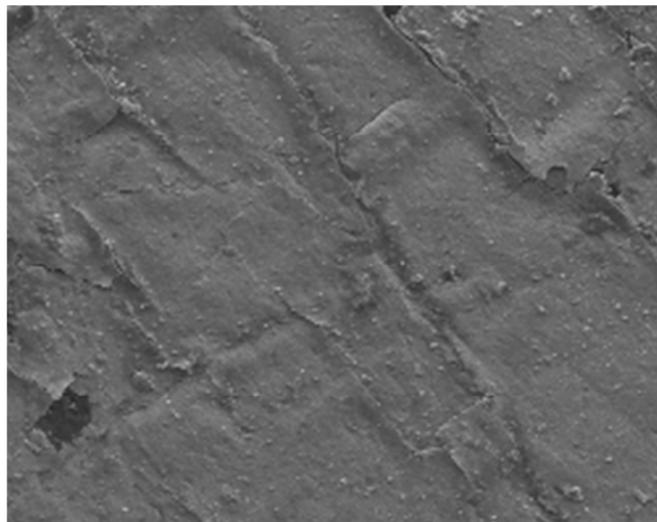


Fig. 7. SEM showing surface cracks on baked (MCS 15) heat treated EN film (x3000 mag.)

3.3. Corrosion Resistance Studies of Mild Carbon Steel Substrates

The photo-macrographs of surfaces of the samples before and after immersion in the medium for 30 days are shown in

Figs. (8a) and (8b-f) respectively, while the results of corrosion rate determination of MCS substrate immersed in the medium by weight loss method are presented in Figs. 9-11.

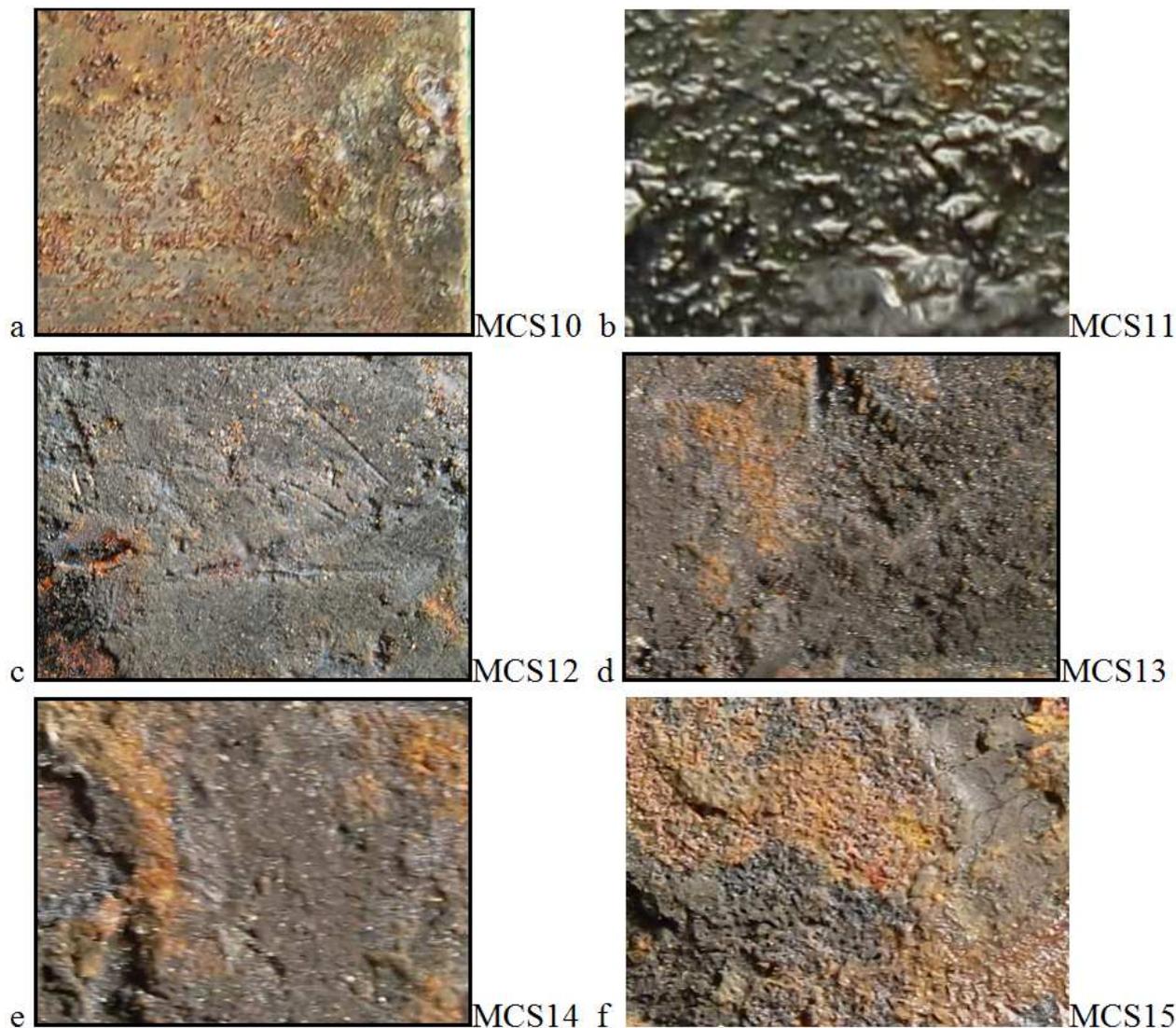


Fig. 8. Photomicrographs of the corroded EN plated MCS samples in 0.5M H₂SO₄ (x50 mag.).

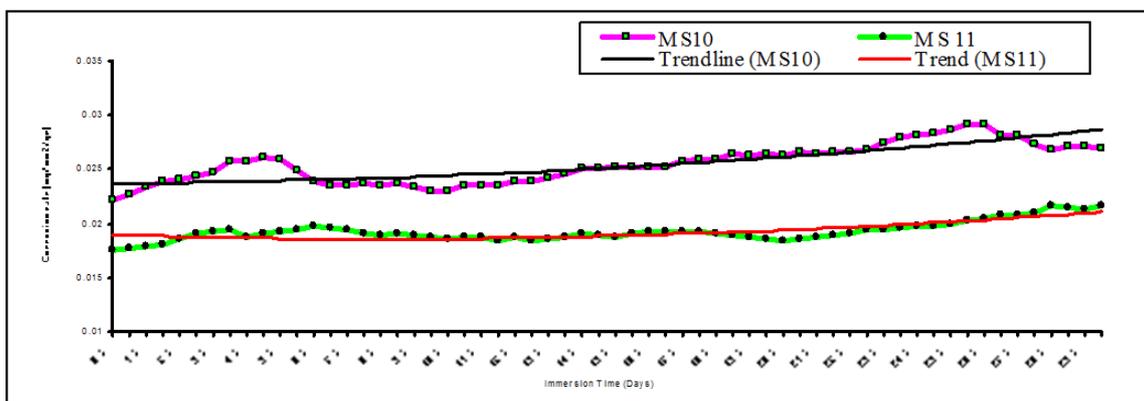


Fig. 9. Corrosion rates (mg/mm²/yr) of MCS10 and MCS11 samples in H₂SO₄.

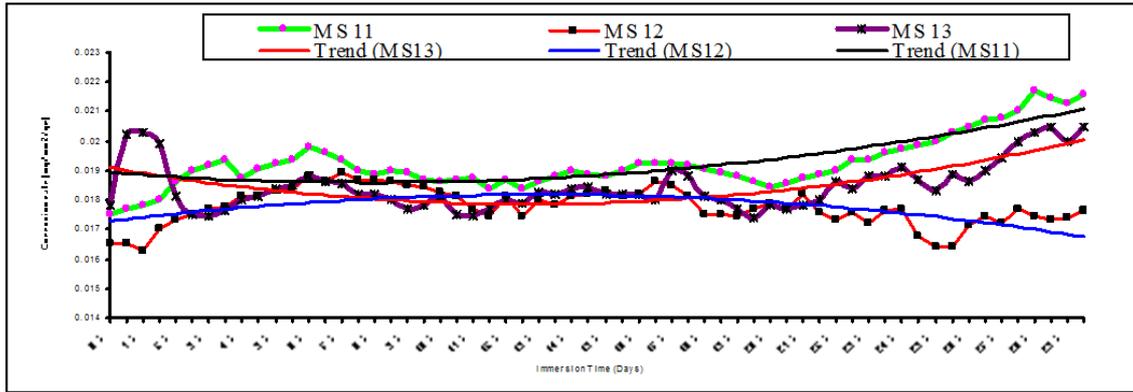


Fig. 10. Corrosion rates ($\text{mg}/\text{mm}^2/\text{yr}$) of MCS11, MCS12 and MCS13 samples in H_2SO_4 .

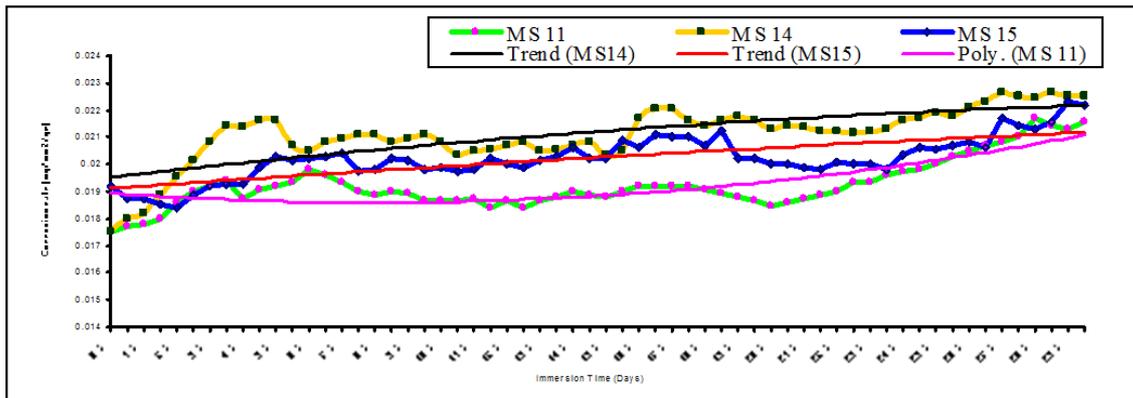


Fig. 11. Corrosion rates ($\text{mg}/\text{mm}^2/\text{yr}$) of MCS11, MCS14 and MCS15 samples in H_2SO_4 .

Fig. 8a shows the photograph of the corroded surface of as received MCS samples (MCS-10) after 30 days of immersion. The corrosion is uniform over the surface. The white patches are the areas that were much affected. The corrosion rate was high for the 30 days of the observation.

Fig. 8b shows the macro-photograph of the corroded EN plated (MCS11) surface after 30 days immersion in the 0.5 M H_2SO_4 solution. Though the corrosion was uniform over the surface, the EN coating makes it less severe in outlook than the corroded surface of the MCS samples. The debris of the surface attack was observed and became more noteworthy after 30 days. The corrosion rate curves of MCS10 (Fig. 8a) and MCS11 (Fig. 8b) samples are illustrated in Fig. 9.

Fig. 8c shows the surface layer of corrosion debris found or deposited on samples MCS12 after the immersion in the solution for 30 days as examined under microscopic camera.

Fig. 8d shows the photo macrograph of corrosion of the MCS13 sample surface after the immersion for 30 days with the waste product left on the samples surface. The colouration signifies the impact of the Fe ion on the corrosion of the base steel material in solution. The trends of the corrosion rates of MCS12 (Fig. 8c) and MCS13 samples (Fig. 8d) are shown in Fig. 10.

Fig. 8e shows the corrosion of MCS14 samples after 30 days with the layers of corrosion debris deposited on the sample surfaces. The brown patches colorations represent the areas of active corrosion at the interface of the electroless-nickel layer and the MCS substrate. The examined surface

under the microscope also revealed the largest amount of pores in comparison to any of the MCS11, MCS12 and MCS13. This is due to the break in the film continuity and poor adhesion of the heat treated EN film to MCS substrate. A large amount of pores were observed on the surfaces after cleaning and examined under the microscope as compared with the MCS10 and MCS11 samples.

This thus allowed the access of the fluid to the MCS substrate surface and corrosion was accelerated.

In Fig. 8f, the surface of the MCS15 sample after the immersion in the medium is shown. The brownish portions show the much large corrosion affected areas as compared with other MCS11, MCS12 and MCS13 sample surfaces in 0.5 M H_2SO_4 . The numerical trends of the corrosion rates of MCS14 (Fig. 8e) and MCS15 (Fig. 8f) samples are illustrated graphically in Fig. 11.

Using the results obtained in Figs. 9-11, the best fit models were developed for the trend using MS excel application of polynomial equations relating the corrosion rates of electroless-nickel in 0.5 M H_2SO_4 . The mathematical model equations were derived for the corrosion rates as follow:

$$R_{\text{MS10}} = 1\text{E-}06x^2 + 1\text{E-}05x + 0.0237 \quad (4)$$

$$R_{\text{MS11}} = 1\text{E-}06x^2 - 5\text{E-}05x + 0.0190 \quad (5)$$

$$R_{\text{MS12}} = -1\text{E-}06x^2 + 7\text{E-}05x + 0.0172 \quad (6)$$

$$R_{\text{MS13}} = 2\text{E-}06x^2 - 0.0001x + 0.0192 \quad (7)$$

$$R_{MS14} = -4E-07x^2 + 7E-05x + 0.0195 \quad (8)$$

$$R_{MS15} = -1E-07x^2 + 4E-05x + 0.0191 \quad (9)$$

Where $0 \leq x \leq 30$, x = immersion time (days), R_{MS10} , R_{MS11} , R_{MS12} , R_{MS13} , R_{MS14} and R_{MS15} are the corrosion rates of the MCS10, MCS11, MCS12, MCS13, MCS14 and MCS15 with respect to immersion time in 0.5 M H₂SO₄ respectively.

Figs. 9-11 compares the corrosion rates (mg/mm²/yr) of the MCS samples (MCS10), EN-plated MCS samples (MCS11) and the four heat treated EN-plated mild steel samples (MCS12, MCS13, MCS14, MCS15) in 0.5 M H₂SO₄. Objectively, the corrosion rate tests were performed to assess the level of surface degradation of the six substrates in 0.5 M H₂SO₄.

The effect of composition of EN coating on the corrosion resistant could be more appreciated from the EDX data obtained (Tables 3-5). The corrosion resistance of an EN coating is a function of its composition as revealed in the EDX data. The EN deposits are unsurprisingly passive (MCS11) and very resistant (MCS12 and MCS13) to corrosion attack as compared with the as-received sample (MCS10) in 0.5 M H₂SO₄ environment. Their degree of passivity and corrosion resistance is greatly affected by the phosphorus content which also influenced the heat treated EN film products.

Predictably, the electroless Ni- P alloys with more than 10 %P is more resistant to the attack in H₂SO₄ environment. Frequently, the vagrant or some co-deposited constituents (such as lead, tin, cadmium, or sulphur) present in the EN play more important roles to its corrosion resistance than the phosphorus content.

One of the principal variables that can affect the corrosion of EN is its heat treatment as revealed in this study (MCS12 and MCS13). With the heat treated EN-plated MCS samples baked for 60 mins at 150 to 210 °C; the EN film adhesion to the substrate is increased and by these treatments hydrogen is relieved from the substrate and the EN deposit [31]. The heat treatment provides a minor amount of co-diffusion between coating and substrate. Hence reduces the corrosion rates.

As nickel-phosphorus deposits are heated to temperatures above 220 °C, nickel phosphide particles begin to form [31] instead the of the more corrosion resistant phosphate, hence reducing the phosphorus content of the remaining EN film material [11]. This reduces the corrosion resistance of the coating as shown in MCS14 and MCS15 samples immersed in the solution. In another case, the particles can also create small active corrosion cells, contributing to the additional destruction of the protective EN film deposit.

It has been reported that EN exposure to temperatures above 220 °C causes reduction in strength and this can destroy the ductility [11, 32, 33, 34, 35]. Usually, hardening type of heat treatments reduce both ductility and the strength of EN deposits, hence the coating breaks and resulted in high corrosion rate.

The contraction of the EN film resulting from the heat treatment (hardening) leads to crack formation on the coating that exposes the steel surface to corrosion attack. These are very much obvious the MCS14 and MCS15 corrosion as shown in Fig. 11. The EN heat treated at high temperature

has tendency to crack under repetitive loads which implies a considerable reduction in the fatigue strength of steel substrates. The behaviour is different from the previous reports in the literature obtained from the EN plating of aluminium alloy [2-4, 26, 36-37] subjected to heat treatment [30] wear [19, 38-40] or corrosion [25, 41] or both [42]. The differences could be attributed to the EN plating bath parameters; mechanical properties, surface morphologies and the chemical compositions of the substrates [2-4, 20, 30, 43-45]. Hence the EN baking at temperatures below 220 °C is encouraged by this study.

4. Conclusion

The collective effects of EN plating and heat treatment on the corrosion of the as-received mild carbon steel substrate, as EN-plated samples and the heat treated EN-film immersed in 0.5 M H₂SO₄ solution have been studied. From the results obtained, it will be right to select EN-plated samples (MCS11) and the low temperature heat treated EN-film (MCS12 and MCS13) for the reason of solving the problem of MCS corrosion in the medium. The application of EN plating enhanced the corrosion resistance of MCS at much lower rate than the heat treated heat treated EN-film (MCS14 and MCS15) in 0.5 M H₂SO₄ environment.

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