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Structural evolution, thermomechanical recrystallization and electrochemical corrosion properties of Ni-Cu-Mg amorphous coating on mild steel fabricated by dual-anode electrolytic processing

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ABSTRACT

The electrolytic Ni-Cu based alloy coating with admixed interfacial blend of Mg have been successfully prepared on mild steel substrate by dual anode electroplating processes over a range of applied current density and dwell time. The electrocodeposition of Ni-Cu-Mg coating was investigated in the presence of other bath additives. The influence of deposition current on surface morphology, adhesion behavior, preferred crystal orientation, surface topography and electrochemical activity of Ni-Cu-Mg alloy coating on mild steel were systematically examined. The thermal stability of the developed composite materials was examined via isothermal treatment. Scanning electron microscope equipped with EDS, X-ray diffraction, Atomic force microscope, micro-hardness tester and 3 μmetrohm Potentiostat/galvanostat were used to compare untreated and isothermally treated electrocodeposited composite. The induced activity of the Ni-Cu-Mg alloy changed the surface modification and results to crystal precipitation within the structural interface by the formation of Cu, Ni_2Mg_3 phase. The obtained results showed that the introduction of Mg particles in the plating bath generally modified the surface and brings an increase in the hardness and corrosion resistance of Ni-Cu-Mg layers fabricated. Equally, isothermally treated composites demonstrated an improved properties indicating 45% increase in the micro-hardness and 79.6% corrosion resistance which further showed that the developed composite is thermally stable.

1. Introduction

The advancement in the field of uniform structural crystal with unique admixed properties by electro-deposition processing has been studied extensively in recent times because of their unique engineering importance [1]. The process and method of electrocodeposition have been widely reported as one of the ways of producing alloy on the surface of metals [2,3], considered to be economical [4] and a convenient route of protection [5]. In particular, multilayered thin films coatings have found useful engineering applications due to their excellent mechanical and corrosion resistance properties. Mild steel have been a candidate material for majority of structural application. This is also coupled with its wide range of industrial application because of its easy availability, ease of fabrication, low cost and good tensile strength among others [6–9]. However, in some service conditions, its functionality and durability are subjects of concern due to corrosion and mechanical deterioration [10]. Many different types of coating are applied commercially to provide protection from corrosive environments and for properties enhancement and thus to extend the life span of mild steel based infrastructures [2]. Though, nickel is anodic to steel, it protects the base metal even when the deposit is porous. However, Ni has limited application as structural materials due to the extremely poor plasticity at room temperature. In order to widen the industrial applications, a significant attention has been paid to amorphous coatings which have the potential to be extensively used in the aggressive environments [1]. Copper is an excellent choice for an under-plate, since it often covers minor imperfections in the base metal. It is relatively inert in most plating solutions of other common metals with high plating efficiency, resulting in excellent coverage even on difficult-to-plate parts. Copper deposits also act as thermal expansion barriers by absorbing the stress pro-

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Table 1
Nominal chemical composition (wt%) of mild steel substrate.

Element	C	Mn	Si	P	S	Al	Ni	Fe
% Composition	0.15	0.45	0.18	0.01	0.031	0.005	0.008	Balance

duced when metals with different thermal expansion coefficients undergo temperature changes, and this is particularly helpful with plastic substrates [11]. A good number of researches have been done extensively using Zn-Ni coating [12–14], nickel with ceramics admixed from single to binary and ternary co-deposition [15] and nano-material [16,17]. More so, bath additives, during electrodeposition further enhance bright bath formulation, good surface appearance and better structural growth [1]. Giz et al. [18] studied the electrochemical behavior Ni-Cu-Fe coatings with excellent results. Therefore, for the drive of improving the oxidation process and hardness of this coating, the combined use of Ni-Cu alloy was proposed using admixed bath constituents coupled with Mg addition (forming a ternary Ni-Cu-Mg alloy) in order to further improve the strengthening effect. Magnesium tends to possess good properties ordinarily but is extremely active which tend to corrode too fast. However, in an admixed bath it has attested to cause preferred deposit orientation which could enhance mechanical crystallization of the coating. This in turn provides better grain size, brightness and reducing internal stress and pitting [19]. Hence, there is limited report on the structural modification and texture formation of Ni-Cu-Mg ternary alloy co-deposited for plating conditions and to be thermo-mechanically studied for further properties enhancement. In this study, an in-depth of the electrolyte bath conditioned with additives on the electro-microstructural and thermo-mechanical properties of Ni-Cu and Ni-Cu-Mg alloy thin films was fabricated. In an event and as a measure of the thermal stability of Ni-Cu-Mg alloy thin films fabricated by electrocodeposition on mild steel, effort was made to thermally treat the coated composite and then re-examine its micro-hardness and corrosion resistance. The investigation attempt to further attest a degree of improvement through harnessing of induced particulate and varied process parameter that is formidable for multilayer growth of Ni-Cu-Mg based alloy for functional and high thermal-oxidation applications for mild steel.

2. Experimental procedure

2.1. Preparation of substrate, coatings and bath constituent's admixture

The dimension of the mild steel (substrate) used was 40 mm × 20 mm × 2 mm sheet and Nickel/Copper sheets of 30 mm × 20 mm × 2 mm were prepared as anodes. The mild steel specimens' chemical composition is shown in Table 1. The cathode was mild steel coupons and anode was commercially pure Ni. The mild steel substrate earlier prepared was actuated by dipping into 10% HCl solution for 10 s followed by rinsing in distilled water. Analar grade chemicals and distilled water were used to prepare the plating solution at room temperature. Prior to plating, the magnesium were added to the prepared Ni-Cu particles electrolytic solution as shown in Table 2. The formulations were then heated to 40 ± 2 °C to ease admix and dissolution of any agglomerate in the bath solution. The prepared Ni-Cu-Mg composite have bath composition as shown in Table 2 and was heated for 2 h and periodically stirred to obtain a homogenous solution before electrolytic deposition process. The prepared cathode and anodes were connected to the D.C. power supply through a rectifier at a varying current of 1.5 and 2 A with an applied voltage of 1.0 V for dwell time of 5 and 10 min. The distance between the anode and the cathode and the immersion depth was kept constant. Thereafter, the samples were rinsed in distilled water and dried.

Table 2
Bath composition of Ni-Cu-Mg alloy co-deposition.

Composition	Mass concentration (g/L)
$CuSO_4$	100
$MgSO_4$	30
Boric acid	10
Glycine	10
Ni_2SO_4	100
pH	4.8
Current	1.5–2.0 A
Time	5, 10 min
Tempt	40 ± 2 °C

Table 3
Formulated bath composition parameter for Ni-Cu-Mg/mild steel composite.

Sample order	Material sample	Time of deposition (min)	Current (A)
Blank	–	–	–
Sample 1	Ni-Cu-Mg	10	2 A
Sample 2	Ni-Cu-Mg	10	1.5 A
Sample 3	Ni-Cu-Mg	5	2 A
Sample 4	Ni-Cu-Mg	5	1.5 A

The formulated bath composition parameter can be found in Table 3.

2.2. Electrochemical measurement and thermal treatment of the composite

The specimens were embedded in epoxy resin leaving a working area of 1.2 cm^2. The working surface was subsequently ground with grinding papers from 600 down to 1800 grit, cleaned by distilled water and ethanol. A conventional three electrode cell, consisting of saturated calomel (SCE), graphite, and coated MS as reference, auxiliary, and working electrode respectively, was used to study the electrochemical behavior of the deposited composite, as-received mild steel and thermally treated composites in 3.65 wt% NaCl solution (The electroform of coated Ni-Cu-Mg-mild steel were heat treated at a temperature of 200 °C for 2 h in an electrical heating furnace under atmospheric conditions and then air cooled). The electrochemical measurement was done with Autolab PGSTAT 101 Metrohm potentiostat/galvanostat. An electrolytic cell containing 50 ml of electrolyte, with and without plated sample, a graphite rod which works as auxiliary electrode and silver chloride electrode (SCE) as reference electrode were used. The potentiodynamic potential scan was fixed to run from −1.5 V to +1.5 mV with scan rate of 0.012 V/s. The electrochemical corrosion test was performed at room temperature in a static solution.

2.3. Hardness measurement and thermal treatment of the composite

The Vickers micro-hardness tester (Dua scan inventor-EMCOTEST) under a load of 0.1 kg with a dwell time of 15 s was applied. Hardness values were taken at four different points and the average used. The indentations started at the surface of the coated-body through the base of the mild steel substrate. For isothermal treatment of the Ni-Cu-Mg/mild steel coated composite, the coated composite samples were isothermally treated and properties were re-examined after thermal treatment. The electroform of coated Ni-Cu-Mg-mild steel were heat treated at a temperature of 200 °C for 2 h in an electrical heating furnace under atmospheric conditions and then air cooled.

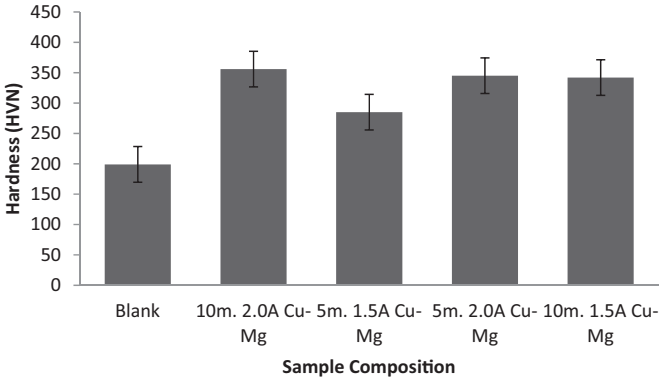

Fig. 1. Variation of micro-hardness and sample composition for the composite.

3. Results and discussion

3.1. Micro-hardness and corrosion characteristics of the developed composite

3.1.1. Micro-hardness studies

In this study, the effects of Ni-Cu-Mg at applied voltage of 1.0 V in 5 and 10 min dwell time with varying current of on mild steel was investigated has shown in Fig. 1. From the result, the micro-hardness of the developed composite increased with additive dispersed into the bath. The pronounced micro-hardness improvement with all samples upon applied current suggests that magnesium (Mg) have a significant strengthening effect. Specifically, the hardness of the coated mild steel before the isothermal treatment increased with an increase in addition of ternary Ni-Cu-Mg alloy and along with the variation of applied current/dwell time. This is similar with earlier reports [4,14,20]. A maximum hardness value of 356HVN was obtained at 2.0 A in 10 min dwell time indicating 44.94% improvement away from uncoated mild steel (Fig. 1). Equally, at 2.0 A for 5 min a higher value of 345HVN was obtained. In general, with 5 min dwell time, even at 2.0 A, the hardness was excellently enhanced (342HVN) indicating 42.7% increment. It is important to note that plating of Ni-Cu-Mg thin film on mild steel substrate can best be optimize at higher current of 2.0 A and enough dwell time of 10 min under the present experimental condition. In addition, the isothermally (tempered) treated composite coatings shows a relatively high hardness value than the un-treated samples. Such improvement observed with the composites can equally be attributed to the intermetallic compounds formed such as Ni_2Mg_3, Cu_2Ni_3Mg which might have refined the structures to smaller one and serves a barrier to dislocation motion giving rise to better hardness.

Specifically, tempered composite at 2.0 A for 10 min have hardness of 362HVN corresponding to 45% improvement. The observation can be ascribed to the refined grains of coatings which gave good interfacial bonding. This occurrence is similar to the results obtained elsewhere [21,22]. The percentage interval between the hardness of the treated and untreated composite was 6%. As a result, it can be said that the tempered composite have a tremendous improvement than that of any composite at all dwell time (Fig. 2). Equally, the SEM micrograph of the composite with the best hardness profile upon addition of Mg (at 2.0 A for 5 min a higher value of 345HVN) indicates a refined surface morphology, which might have led to its better hardness value than other composites (Fig. 6a,b). The significant increase in the micro-hardness with all sample upon conditioned plating parameter, suggests that magnesium have a unique strengthening effect. These were believed to have altered the morphology and caused grain refinement, hence, providing structural modification which

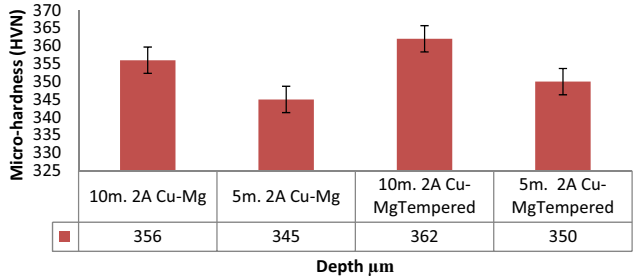

Fig. 2. Variation of micro-hardness for the as-deposited and heat-treated conditions.

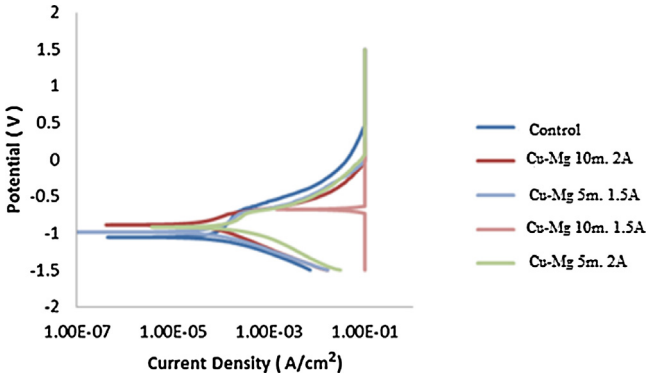

Fig. 3. Linear polarization curve for the coated and un-coated mild steel samples in 3.65% NaCl solution.

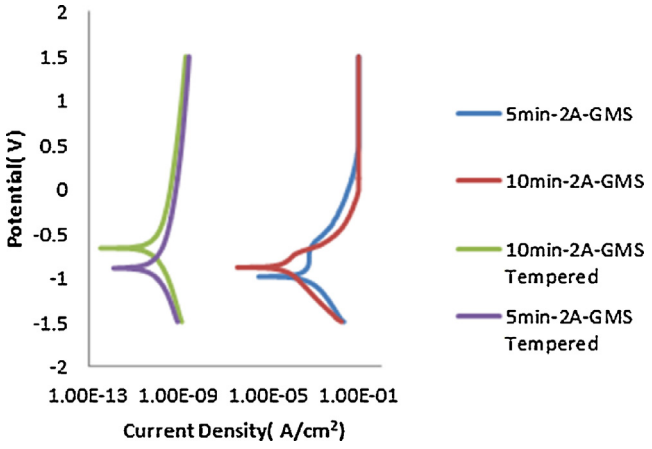

Fig. 4. Linear polarization curve for the as-deposited and heat treated conditions.

improved the hardness of the composite coatings generally. Similar claims have been put forward elsewhere [1,23].

3.1.2. Corrosion characteristics of the developed composite

The corrosion resistance of the developed composite increased with Mg addition, change in applied current and dwell time (Table 4). The composite with dwell time of 10 min and 2.0 A applied current have the best corrosion resistance, which is similar to the results of the micro-hardness of the composite (Fig. 3). An improvement of 79.6% corrosion resistance was achieved (Table 4) indicating that the coatings have enhanced the corrosion performance of the developed composite, with similar report by Hedge et al. [14]. On the other hand, upon isothermal treatment of the composite, there exists a higher improvement in the corrosion resistance of the composite at all dwell time and applied current (Fig. 4 and Table 5). Specifically, treated-composite with 10 min

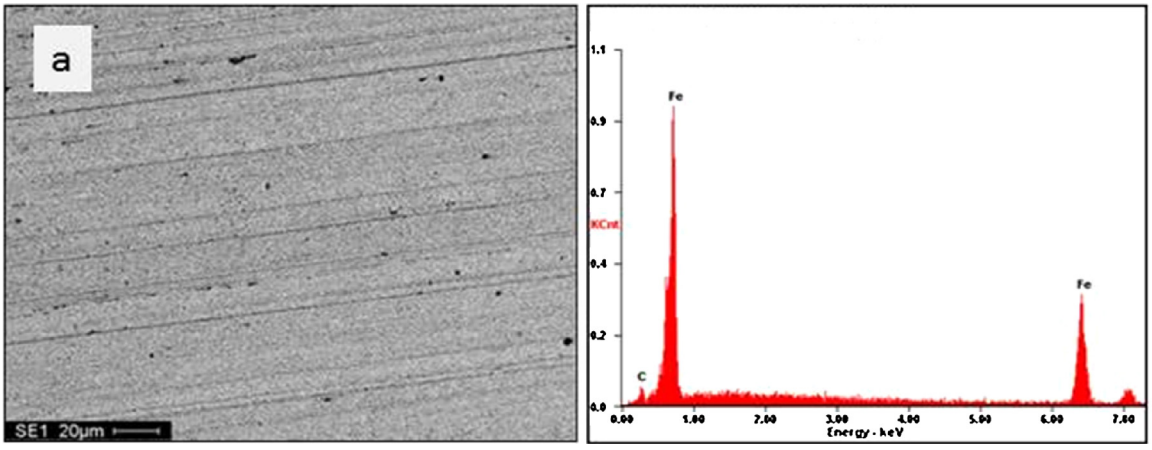

Fig. 5. SEM/EDS micrographs of as-received sample.

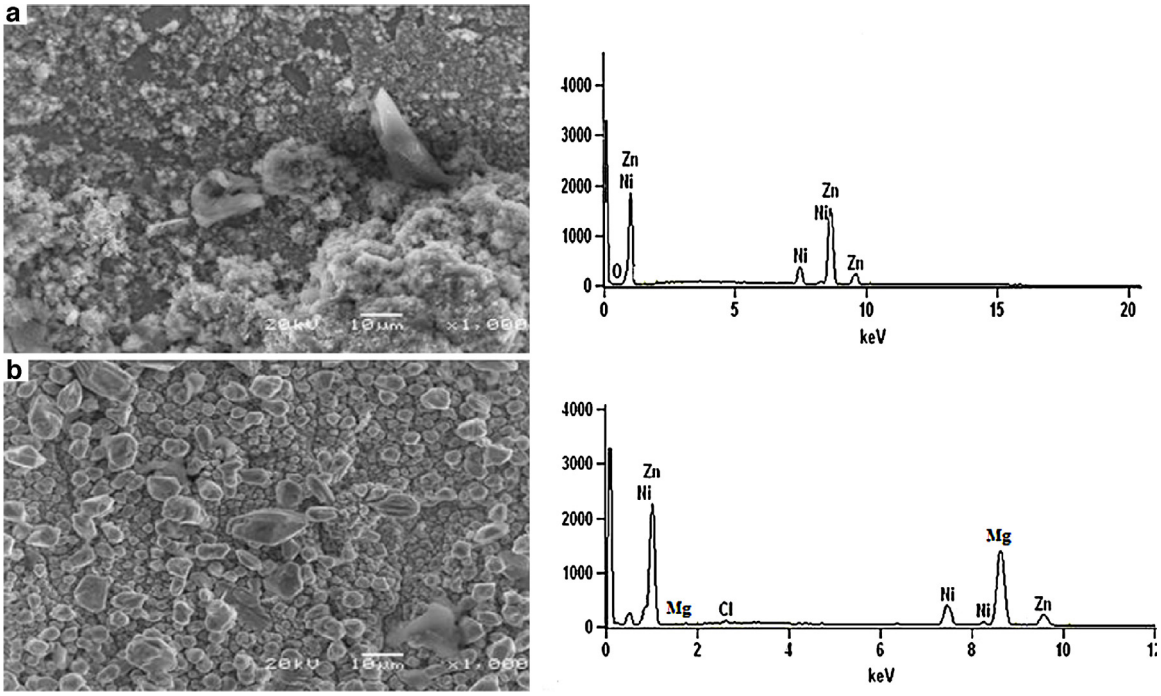

Fig. 6. SEM/EDS micrographs of (a) Ni-Cu at 2 A for 5 min (b) Ni-Cu-Mg at 2 A for 5 min.

Table 4
Polarization data for the developed composite at different conditions in a 3.65% NaCl static solution.

Sample	Ecorr, Obs (V)	jcorr (A/cm^2)	icorr (A)	Cr (mm/year)	Rp (Ω)
10 min 1.5 A GMS	−0.89970	2.38×10^{-5}	2.38×10^{-5}	0.24498	519.94
10 min 2.0 A GMS	−0.88433	8.94×10^{-6}	8.94×10^{-6}	0.10388	1718.8
5 min 1.5 A GMS	−0.95230	2.55×10^{-5}	2.55×10^{-5}	0.32002	417.31
5 min 2.0 A GMS	−0.89303	2.11×10^{-5}	2.11×10^{-5}	0.23573	522.16
Control	−1.05340	4.38×10^{-5}	4.38×10^{-5}	0.50938	125.10

Table 5
Polarization data for the untreated and thermally treated composite in a 3.65% NaCl static solution.

Sample	Ecorr, Obs (V)	jcorr (A/cm^2)	icorr (A)	Cr (mm/year)	Rp (Ω)
10 min 2.0 A GMS	−0.88433	8.94×10^{-6}	8.94×10^{-6}	1.04×10^{-1}	1.72×103
5 min 2.0 A GMS	−0.89303	2.11×10^{-5}	2.11×10^{-5}	2.36×10^{-1}	5.22×102
10 min 2.0 A tempered	−0.66783	4.38×10^{-11}	4.38×10^{-11}	5.09×10^{-7}	3.32×108
5 min 2.0 A tempered	−0.89297	4.50×10^{-8}	4.50×10^{-8}	5.23×10^{-4}	7.65×106
Control	−1.05340	4.38×10^{-5}	4.38×10^{-5}	5.09×10^{-1}	1.25×102

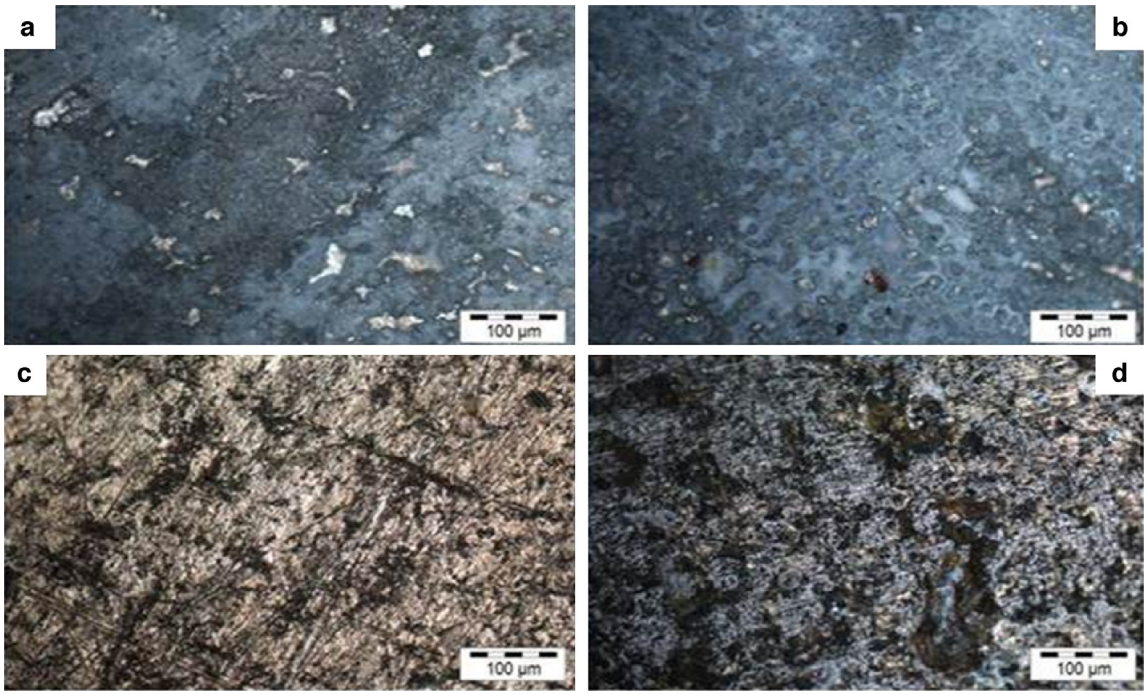

Fig. 7. Optical Structural of (a) 10 min, 2 A-gms-before heat treatment (b) 10 min, 2 A, gms-after heat treatment (c) 5 min, 2 A-before heat treatment (d) 5 min, 2 A-after heat treatment.

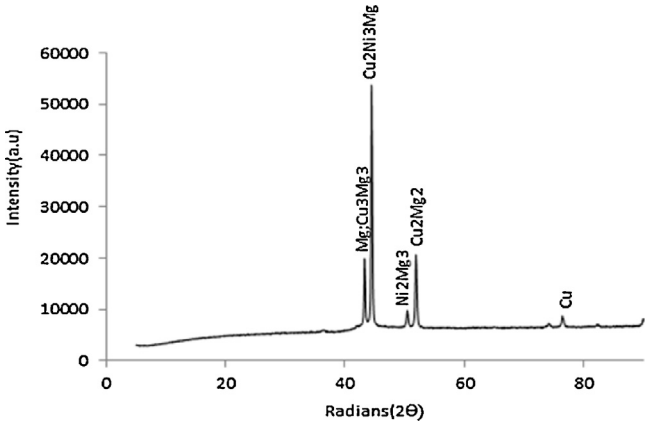

Fig. 8. XRD spectrum of Ni-Cu-Mg coated mild steel at 2 A for 5 min.

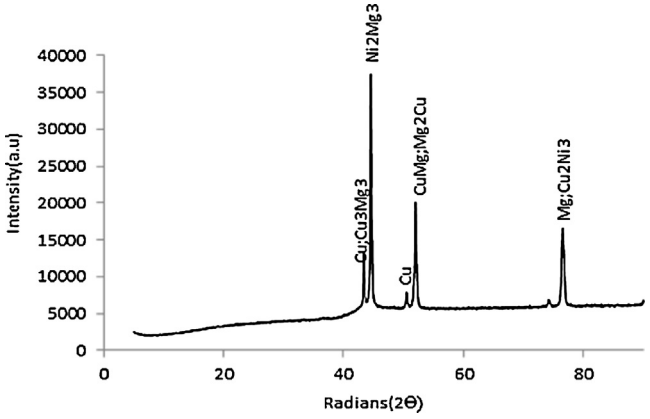

Fig. 9. XRD spectrum of Ni-Cu-Mg coated mild steel at 2 A for 10 min.

and 2.0 A parameter have the least oxidation rate, thus with 99.99% improvement to corrosion at high temperature.

The corrosion rate in the tempered 10 min and 2.0 A condition was 5.09×10^{-7} mm/year (heat treated) and 1.04×10^{-1} mm/year (un-heat treated) with similar trend in the polarization resistance (Table 5).

The corrosion rate and current density decreased significantly. It can be said that the developed ternary coatings reduced the corrosion rate of the mild steel with increased polarization resistance value. This indicates that the admixed coatings matrixes created a good bonding and adhesion with the substrate material. The Ni-Cu-Mg coatings on the mild steel acts as a significant surface resistance to corrosion process which in turn lower the corrosion rate. It can be observed that the developed composite that are isothermally treated have distinct corrosion behavior: lower current density and corresponding higher corrosion potentials (Fig. 4). However, the oxidation resistance of the treated composites was favored positively.

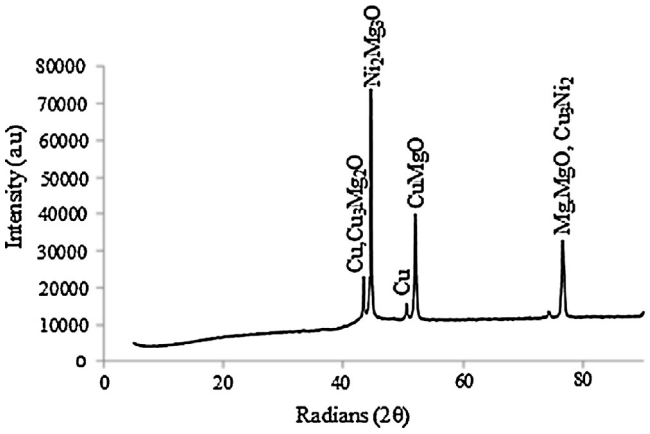

Fig. 10. XRD spectrum of thermally treated Ni-Cu-Mg coated mild steel at 2 A for 10 min.

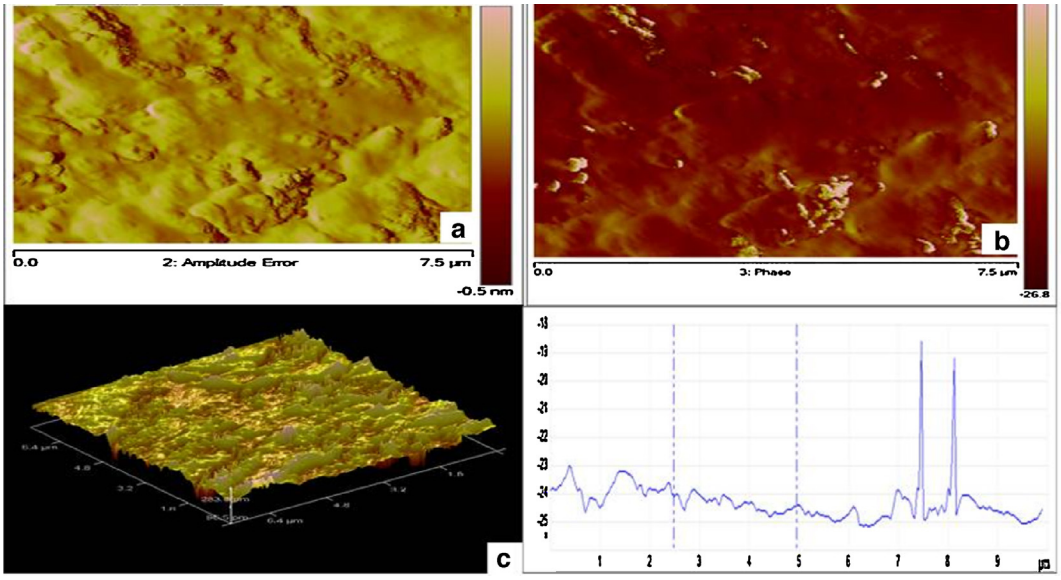

Fig. 11. AFM spectra showing the three dimensional image and the topography of Ni-Cu-Mg at 2 A for 5 min.

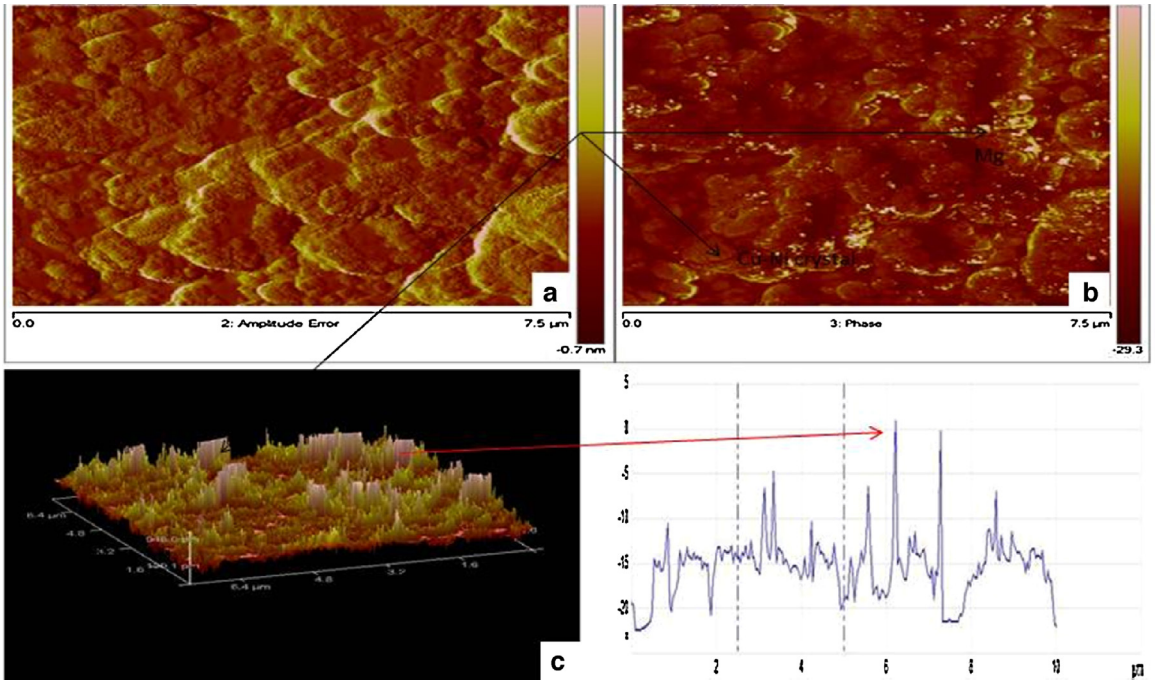

Fig. 12. AFM spectra showing the three dimensional image and the topography Ni-Cu-Mg at 2 A for 10 min indicating the surface interaction and crystal growth.

3.2. SEM surface morphology, strengthening phases and topographical studies of the coatings

3.2.1. SEM surface characterization of the composite

Fig. 5 shows the microstructure of as-received mild steel (substrate). In Fig. 6a, 2.0 A for 5 min, there seems to an incomplete and non refined surface as compared when Mg was added to Ni-Cu alloy to for Ni-Cu-Mg ternary films (Fig. 6b). The surface of the coat containing Ni-Cu-Mg alloy on mild steel have sufficiently demonstrated a more coherent solute atoms and grain refinement which might have accounted for the improved properties. Equally, the coating does not contain crack and the Mg addition has improved the uniformity and crystal growth of the ternary-coated body. In the thermally treated (Fig. 6b,d) and untreated (Fig. 7a,c) conditions, the surface morphology of the samples generally indicated an oxidized surface (Fig. 7b,d). This however calls for further investigation since it is expected that such surfaces may likely have contained un-adhenced film coating at that temperature and probably it may undergo scaling since oxidation is likely to occur. Meanwhile the explanation for such occurrences is not very clear. Similar reports have been put forward in our previous study [10,24]. However, it can be said that the film formed on the mild steel substrate are isothermally stable, this is however, a measure of thermal stability rather than extensive study of thermal analysis. Hence the composites indicate that it responded positively to the bath admixture during the electrocodeposition of the coatings.

3.2.2. XRD phase evaluation and atomic force microscopy (AFM) analysis

Figs. 8 and 9 show the X-ray diffraction spectra of some coated samples with different phases. For example in Fig. 8, the predominant phases are Cu_2Ni_3Mg, Cu_2Mg_2, Ni_2Mg for electrocodeposition

at 2.0 A with 5 min. Similarly, in Fig. 9, a hard phase of Ni_2Mg_3 coupled with CuMg, Mg_2Cu, Cu_2Ni_3, Cu_3Mg_3 phases are formed besides original Cu/Ni based particulate which was observed within coating interface. These might have promoted the strengthening behavior of the composite at 2.0 A with 5 min plating condition. Higher peak of the resulting phases are as a results of the admixed particulate of Mg incorporated that resulted into co-deposition and in turn led to formation of high strength composite matrix. This was evidenced from the EDS of Fig. 2b when Mg was inoculated forming Ni-Cu-Mg ternary based bath. However, upon thermal treatment of the Ni-Cu-Mg coated mild steel at 2 A for 10 min sample there exists formation of oxide based phases such as MgO, Cu_3Ni_2, Cu_3Mg_2O on the surface of the treated samples. The interaction between Ni-Cu and Mg ultimately resulted into a crystal orientation of grain size rather than chemical dissolution. Equally, the improvement of different crystal structures can be related to the growth of grains due to its compactness. The elemental detection from the EDS relate with the strengthening phases observed in the XRD spectral in all the composites produced. Generally, all the deposited samples provided a good reflection of surface structure and texture expected within the composite microstructure even after thermal treatment (Fig. 10).

AFM of Ni-Cu-Mg films obtained at 1.0 V for 5 and 10 min deposition condition are shown in Figs. 11 and 12. In Fig. 11, little or no crystal growth can be seen, which might be that the dwell time does not give sufficient plating and enough time for complete crystal growth. While in Fig. 12, the topography corresponds to the improved properties of the composite obtained with 10 min at 2.0 A applied current. These show that a uniform crystallites coalesced along with smaller grains were found affirming the result obtained from scanning electron micrograph. With Fig. 12, the crystal growth and orientation are slightly uniform and Cu/Ni crystal is distinct along with Mg and other inoculants additive. These indicate a smother and uniform topography of the Ni-Cu-Mg film on the mild steel substrate which demonstrated better adhesion of the resultant coatings. This also affirmed the result obtained for this composite at the plating conditions.

4. Conclusions

The surface of the coat containing Ni-Cu-Mg alloy on mild steel have sufficiently demonstrated a more coherent and grain refinement which might have account for the improved properties. The thermal stability of already developed Ni-Cu-Mg thin films on mild steel by electrocodeposition was evaluated with the aim of examining its micro-hardness and corrosion resistance in static sodium chloride solution before and after isothermal treatment. In the event, the samples were further subjected to thermal treatment at 200 °C for 2 h in an electrical heating furnace under atmospheric conditions and then air cooled. A maximum hardness value of 356HVN was obtained at 2.0 A in 10 min dwell time indicating 44.94% improvement away from uncoated mild steel. Equally, at 2.0 A for 5 min a higher value of 345HVN was obtained while the oxidation resistance rather favored the composite developed. The corrosion resistance of the developed composite increased with Mg addition, change in applied current and dwell time with an improvement of 79.6% corrosion resistance achieved. Equally, the predominant phases are Cu_2Ni_3Mg, Cu_2Mg_2, Ni_2Mg in untreated and Cu_3Mg_2O, MgO phases the treated sample as studied by the in situ XRD investigation. There was an indication of a smother and uniform topography of the Ni-Cu-Mg film on the mild steel substrate resulting in better adhesion of the resultant coatings.

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