

Research Article

Electroless Deposition of Ni-Cu-P Coatings Containing Nano- Al_2O_3 Particles and Study of Its Corrosion Protective Behaviour in 0.5 M H_2SO_4

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Ni-Cu-P/nano- Al_2O_3 composite coatings are prepared on mild steel from an alkaline electroless plating bath containing different concentrations of Al_2O_3 nanoparticles. The protective effect of codeposited nanoparticles on the corrosion behaviour of the coatings is studied in 0.5 M H_2SO_4 solution. The electrochemical methods, that is, electrochemical noise (ECN), electrochemical impedance spectroscopy (EIS), and polarization measurements, are used to characterize the corrosion properties of the coatings. The results show that the inclusion of nanosized particles leads to significant improvement of corrosion resistance of the coatings. The highest corrosion resistance is obtained at 20 ppm of nanoparticles concentration in the plating bath. The ECN measurements results are in good agreement with results obtained from two other electrochemical methods after trend removal. The SEM images prove that nano- Al_2O_3 particles were embedded in the Ni-Cu-P matrix and are dispersed uniformly on the coating surface.

1. Introduction

Electroless nickel plating is an autocatalytic process which has found broad applications in industry due to attractive properties of resulting metal coatings, such as excellent corrosion protection properties, wear resistance, deposit uniformity, and solder ability [1–5]. After extending to commercial applications, electroless nickel plating has flourished rapidly and now is a constituted industrial process. Some ternary electroless nickel alloys coatings such as Ni-Cu-P, Ni-Co-P, Ni-Cr-P, and Ni-Mo-P were prepared and reported as barrier corrosion protection coatings [6].

Although electroless Ni-P coatings can serve many roles, the search to improve coatings properties, such as hardness, antiwear, and corrosion protective performance, has led to getting incorporation of many solid particles in the matrix of the electroless Ni-P alloy coatings. These solid particles can be hard materials (such as SiC, Al_2O_3 , SiO_2 , and diamond) to enhance the hardness and/or wear resistance of the deposits or they can be dry lubricants (such as MoS_2 , PTFE, and

graphite) to impart lubricity and to reduce the friction coefficient [7]. The choice of the type of particles depends on the specific properties desired. Therefore by selection of suitable fine particles, composite coatings with highly specific characteristics can easily be produced [8].

The different properties of electroless plating Ni-P coatings such as protective performance have been investigated extensively [9]. But little corrosion investigated data are available for the composite coatings based on Ni-P. The composite coatings such as Ni-P-PTFE, Ni-Cu-P, and Ni-Cu-P-PTFE were prepared by electroless deposition and their corrosion performance in HCl and NaCl solutions has been studied in detail [6, 10, 11]. Codeposition of copper in Ni-P matrix has unfathomable effect on deposit properties such as corrosion-resistance [12]. In the presence of a little amount of copper, smoothness, brightness, ductility, and corrosion resistance of the electroless Ni-P deposit increase enormously [13, 14]. Nanosized particles such as Al_2O_3 [15–17], WC [18], and TiO_2 [19] are also used to improve some surface properties of Ni-P electroless coatings such as wear resistance and hardness.

There are several conventional methods such as electrochemical impedance spectroscopy (EIS) [20], polarization [21], and electrochemical noise (ECN) [22] measurements that are employed to investigate corrosion performance. Among these techniques, ECN method has some outstanding advantages over other electrochemical methods. These advantages include low cost of the equipment, ease of data collection, and lack of the need for imposing any perturbation on the electrochemical system, thus maintaining the sample surface intact and categorise as nondestructive procedure [23, 24]. However, because of the novelty, complexity of data analysis, and sensitivity to environmental noises, this approach has received little attention with respect to the other techniques in the corrosion studies [25, 26].

In the present research, Ni-Cu-P ternary and Ni-Cu-P-nanoAl₂O₃ quaternary alloys were prepared by electroless plating method. In the case of quaternary composite coatings preparation, the different amounts of nano-Al₂O₃ particles were added to the electroless baths. The protective effect of nano-Al₂O₃ particles content on the corrosion behaviour of alloy coatings was studied using electrochemical methods including EN, EIS, and polarization measurements in the 0.5 M H₂SO₄.

2. Experimental

2.1. Sample Preparation and Pretreatment. Mild steel sheets (1 cm × 1 cm × 0.2 cm) were mounted in polyester resin in such a way that only one side of samples (with a surface area of 1 cm²) was in contact with the bath solution. The samples were mechanically polished with emery papers up to 2000 grade, then cleaned in an alkaline solution (1M KOH) for 15 minutes at room temperature, and successively rinsed with the sufficient deionized water. In order to activate the electroless plating process, samples were immersed in the diluted plating bath (10 times) and a potential of -2 V versus saturated calomel electrode (SCE) was applied using an AUTOLAB PGSTAT30 potentiostat-galvanostat.

The electrolytic nickel strikes were used to activate the electroless plating process by using the 10 times diluted plating bath at a constant potential of -2 V versus saturated calomel electrode. In this way a thin layer of active nickel was deposited and provided a catalytic surface for the preparation of the electroless nickel process. An alkaline electroless plating bath as a base one was used and its chemical composition is given in Table I. Quaternary composite coatings were precipitated from the base electroless bath containing 5, 10, 20, 50, 100, and 1000 ppm of nano-Al₂O₃ particles (50 nm). All chemicals used were of analytical grade and double distilled water was applied for preparation of all solutions. During plating, the temperature of bath was maintained at 85 ± 1°C using a MEMERT thermostat. In each experiment 200 mL of fresh bath solution was selected.

2.2. Electrochemical Methods

2.2.1. Electrochemical Impedance Spectroscopy. The corrosion behaviour of coatings was studied by electrochemical impedance spectroscopy (EIS) technique in 0.5 M H₂SO₄

TABLE I: Chemical composition of electroless plating bath.

Substance	Concentration (g/L)
NiSO ₄ ·2H ₂ O	25
NaH ₂ PO ₂	25
Sodium acetate	20
Sodium citrate	60
CuSO ₄	1
Thiourea	0.001
Surfactant	0.003
pH = 10 ± 0.2	

solution at 25°C. A conventional three-electrode cell was used for EIS studies which consists of an electroless plated sample as the working electrode, a SCE as reference electrode, and a platinum rod as counter electrode [27]. Before each EIS test, the sample was immersed in 0.5 M sulphuric acid solution for 20 minutes at room temperature. The measurements were carried out at open circuit potential by applying a sine wave with amplitude of 10 mV over a frequency range between 0.1 Hz and 10 kHz.

2.2.2. Polarization. The polarization experiments were performed with an AUTOLAB potentiostat-galvanostat (PGSTAT30). Prior to each experiment, the specimen was immersed in 0.5 M H₂SO₄ solution for 20 minutes. The applied potential range was ±250 mV with respect to OCP potential with a scan rate of 2 mV/s. The obtained polarization data were analysed by GPES to obtain corrosion parameters including corrosion potential, E_{corr} , polarization resistance, R_p , and corrosion current density, I_{corr} .

2.2.3. Electrochemical Noise Analysis. Electrochemical noise data were obtained using an Autolab PGSTAT30 potentiostat-galvanostat with GPES (General Purpose Electrochemical Software) version 4.9.005 Beta software. A three-electrode cell (Figure 1) was used that consists of two nominally identical mild steel electrodes as dual working electrodes (each with 1 cm² surface area) and saturated calomel electrode (SCE) as reference electrode. The electrochemical current noise was measured between two working electrodes (coupled with zero resistance ammeter) and, simultaneously, the potential noise of coupled electrodes was measured with respect to the SCE reference electrode [21, 28]. EN measurements were performed just after immersion of steel samples in 0.5 M H₂SO₄. Two points per second ($\Delta t = 0.5$ s) were recorded during the first 256 seconds of immersion. The frequency domain corresponding to the sampling conditions was evaluated to be between $f_{\text{max}} = 1/2\Delta t$ and $f_{\text{min}} = 1/N\Delta t$, where Δt and N are sampling interval and total number of data points, respectively [29]. All experiments were carried out in open circuit potential at 25°C.

3. Results and Discussion

3.1. Electrochemical Impedance Spectroscopy. The Nyquist plots for coatings obtained from electroless plating baths

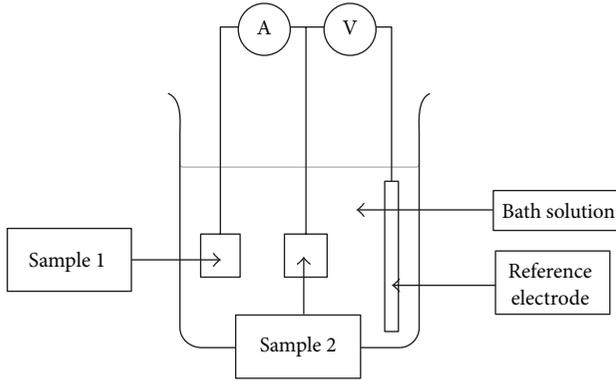
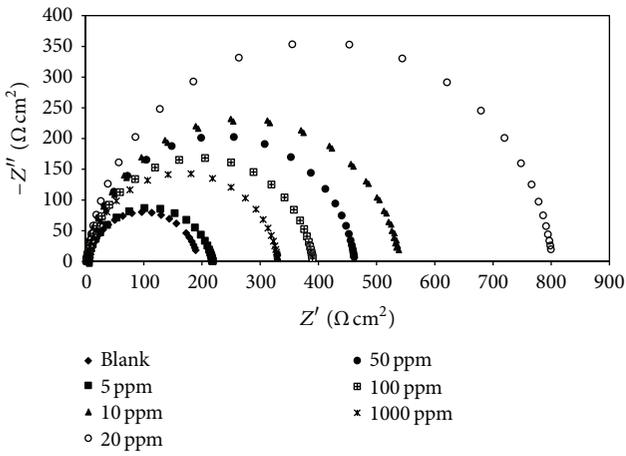


FIGURE 1: Setup for electrochemical noise measurements.


 FIGURE 2: Nyquist plots for composite coatings obtained from plating bath containing different concentration of nano- Al_2O_3 particles.

containing various amounts of Al_2O_3 (0–1000 ppm) are shown in Figure 2. Table 2 gives the various parameters such as charge-transfer resistance (R_{ct}), constant phase element (CPE), and the parameter n .

It is obvious from Figure 2 that the value of charge transfer resistance (R_{ct}) of composite coating obtained from the diameter of semicircles of Nyquist plots, as suggested by Tsuru et al. [30], increases significantly by adding nano- Al_2O_3 particles to the plating bath. The corrosion parameters values such as R_{ct} and CPE were determined by fitting of the impedance data in a Randles equivalent circuit model [31]. The CPE values are obtained at the frequency (f_{\max}) in which the imaginary component of the impedances is maximum ($-Z_{\text{im}(\max)}$):

$$C_{dl} = \frac{1}{2\pi f_{\max} R}. \quad (1)$$

The results show that the presence of Al_2O_3 improves the corrosion resistance of Ni-P coating in 0.5 M H_2SO_4 solution. Adding the amount of Al_2O_3 from zero to 20 ppm in the plating bath increases the surface resistance from 190.4 to 799.9 $\Omega \text{ cm}^2$. Another increase in the alumina concentration

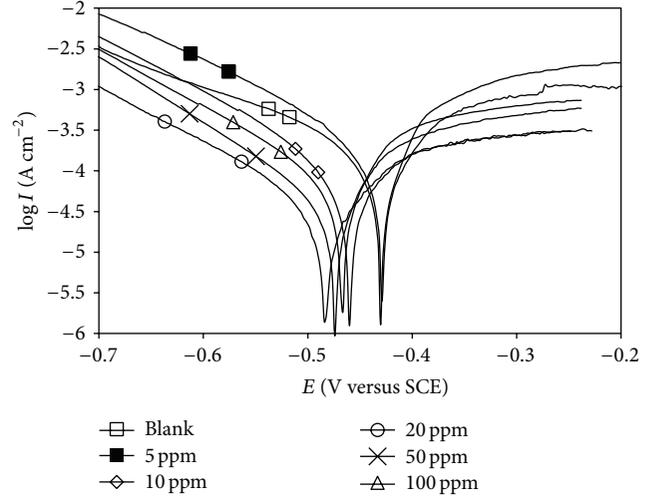

 FIGURE 3: Polarization plots for composite coatings obtained from plating bath containing different concentration of nano- Al_2O_3 particles.

 TABLE 2: Corrosion parameters of coatings in 0.5 M H_2SO_4 derived from EIS measurements.

Concentration of nanoparticles in the bath (ppm)	R_s ($\Omega \text{ cm}^2$)	R_{ct} ($\Omega \text{ cm}^2$)	CPE-Y ($\Omega^{-1} \text{ cm}^{-2}$)	n
0	3.95	190.4	8.35×10^{-5}	0.89
5	2.36	216.0	9.02×10^{-5}	0.86
10	1.95	388.9	5.06×10^{-5}	0.91
20	2.19	799.9	4.77×10^{-5}	0.93
50	2.12	536.0	7.22×10^{-5}	0.93
100	2.01	393.1	6.89×10^{-5}	0.91
1000	2.20	328.6	7.36×10^{-5}	0.91

(up to 1000 ppm) affects negatively the corrosion resistance with respect to low concentrations of nanoparticles, so that the corrosion resistance of the coating obtained at a concentration of 1000 ppm alumina in the bath decreased to about 315.1 $\Omega \text{ cm}^2$.

3.2. Polarization Studies. Figure 3 indicates polarization curves for the coatings obtained from baths containing and without the nano- Al_2O_3 particles in 0.5 M sulphuric acid solution. The corrosion parameters including corrosion potential, E_{corr} , cathodic and anodic Tafel slopes, b_c and b_a , polarization resistance, (R_p), and corrosion rate, (C.R), were obtained from polarization curves and were presented in Table 3. In good agreement with EIS results, nano- Al_2O_3 particles addition to plating bath led to an increase in the corrosion resistance and therefore a decrease in corrosion rate. The corrosion potential did not significantly change for the coatings containing various amounts of nanoparticles. As obtained from the EIS results, the highest corrosion resistance

TABLE 3: Corrosion parameters of coatings in 0.5 M H₂SO₄ derived from polarization measurements.

Concentration of nanoparticles in the bath (ppm)	E_{corr} (mV versus SCE)	R_p ($\Omega \text{ cm}^2$)	b_a (mV/dec)	b_c (mV/dec)	C. R. (mm/year)
0	-430	178.8	61	117	1.14
5	-428	194	86	97	1.20
10	-460	362.3	175	123	1.02
20	-484	790.9	112	114	0.36
50	-473	546.1	90	101	0.44
100	-467	352.3	85	127	0.74
1000	-464	315.3	90	106	0.79

observed from polarisation curve for the coating resulted from an electroless bath containing 20 ppm nano-Al₂O₃ particles.

3.3. Electrochemical Noise Data Analysis. The electrochemical noise measurement is another electrochemical method, which is used in corrosion studies. This technique describes the low level spontaneous fluctuations of potential and current that occur during an electrochemical process like corrosion phenomena. The electrochemical noise provides a possible way to calculate a special form of polarization resistance, namely, statistical polarization resistance or noise resistance (R_n). Noise resistance is defined as follows [32]:

$$R_n = \frac{\sigma_V}{\sigma_I}, \quad (2)$$

where σ_V and σ_I are the standard deviations of potential and current noise data, respectively.

Voltage and current noise data are regarded as random fluctuations around some mean values. In the case of voltage noise, the mean value is the steady state potential. Steady state potential tends to drift during the electrochemical process and it was shown that this drift can greatly influence the results obtained from an analysis of the ECN data. The phenomena are referred to as DC trend and the process of removing it is called trend removal [33]. There are some mathematical methods for trend removal of random ECN data. In this experimental work, the MAR method was used. This method is briefly explained as follows [34].

Suppose that a series of voltage noise-time records, $\{V_n\}$, $n = 1, 2, 3, \dots, i, i + 1, \dots, k$, is experimentally recorded. Any individual data point in the group $\{V_n\}$, V_i , is a combination of the random noise component and the DC trend component which are functions of time, t :

$$V_i(t) = V_{i,\text{noise}}(t) + V_{i,\text{DC}}(t). \quad (3)$$

$V_{i,\text{noise}}(t)$ is the real noise and is required for future noise analysis. $V_{i,\text{DC}}(t)$ is the DC trend component that must be

TABLE 4: Noise parameters of coatings in 0.5 M H₂SO₄ derived from electrochemical noise measurements.

Concentration of nanoparticles in the plating bath (ppm)	σ_V (V)	σ_I (A cm ⁻²)	R_n ($\Omega \text{ cm}^2$)
0	2.00×10^{-5}	9.94×10^{-8}	201
5	1.59×10^{-4}	7.34×10^{-7}	217
10	5.75×10^{-5}	1.38×10^{-7}	416
20	9.44×10^{-5}	1.19×10^{-7}	794
50	2.42×10^{-5}	4.40×10^{-8}	549
100	1.28×10^{-4}	3.57×10^{-7}	360
1000	3.92×10^{-5}	1.24×10^{-7}	317

removed. A good estimation of $V_{i,\text{DC}}(t)$ can be obtained from an average value of adjacent data points of V_i :

$$V_{i,\text{DC}}(t) = V_i(p) = \frac{\sum_{i-p}^{i+p+1} V_i}{(2p+2)}, \quad (4)$$

where p can be 1, 2, 3, or more. $V_{i,\text{noise}}$ can be obtained as

$$V_{i,\text{noise}}(t) = V_i(t) - V_{i,\text{DC}}(t). \quad (5)$$

The average voltage changes for each recorded point. The noise signals fluctuate around these average values. The same method can also apply for current noise. We wrote a suitable computer program in Matlab 6.5 software for trend removal process based on MAR method. P value [34] was 3 during this process.

Figure 4 shows some of the electrochemical potential noise data before and after trend removal for the coatings containing and without nano-Al₂O₃ particles. Some of the current noise diagrams have also been shown in Figure 5. Before trend removal, deviation from average baseline is observed. The data including potential and current standard deviations and noise resistances in different prepared coatings are presented in Table 4. As the EIS and polarization studies, the ECN results show that the noise resistance,

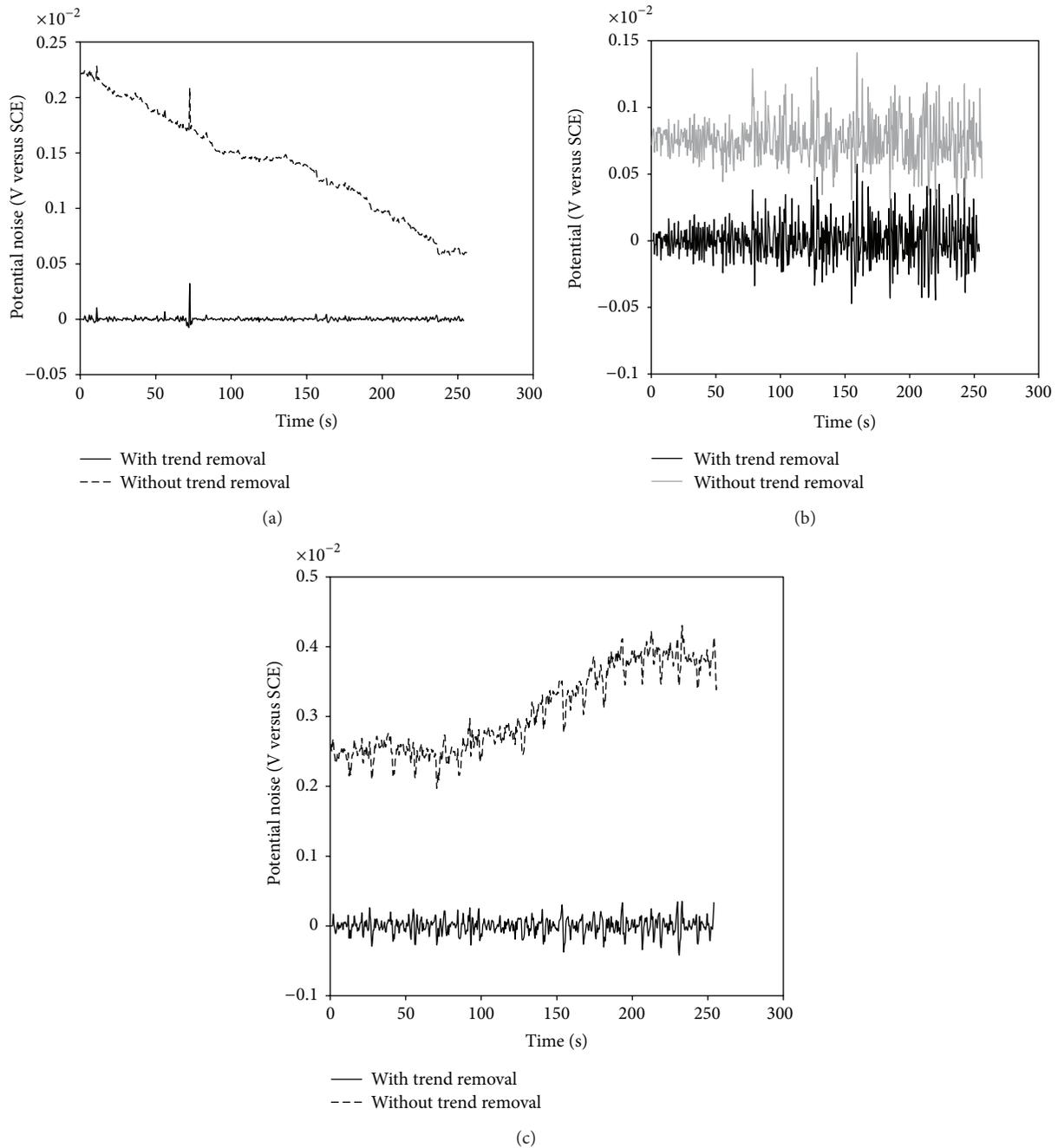


FIGURE 4: Electrochemical potential noise diagrams before and after trend removal for composite coatings obtained from plating bath containing different concentration of nano- Al_2O_3 particles: (a) blank, (b) 5 ppm, and (c) 20 ppm.

as the specific type of polarization resistance, significantly increases in the presence of different concentrations of nanoparticles. As it can be observed, ECN results after trend removal are in good agreement with EIS and polarization data. Optimum concentration of nanoparticles in the corrosion resistance improvement was found to be 20 ppm. These results also proved that electrochemical noise method is a suitable method to study the corrosion rate of the coatings.

In general, electroless Ni-P as a barrier film protects the substrate from the corrosive environments by sealing it. High resistance of the amorphous Ni-P coats is a direct result of nature and passivity of the Ni-P deposits. The amorphous alloys offer better corrosion resistance than equivalent polycrystalline materials, due to freedom of grain or grain boundaries as well as glassy nature [6, 35]. It is evident from literature reports on Ni-P coatings that preferential

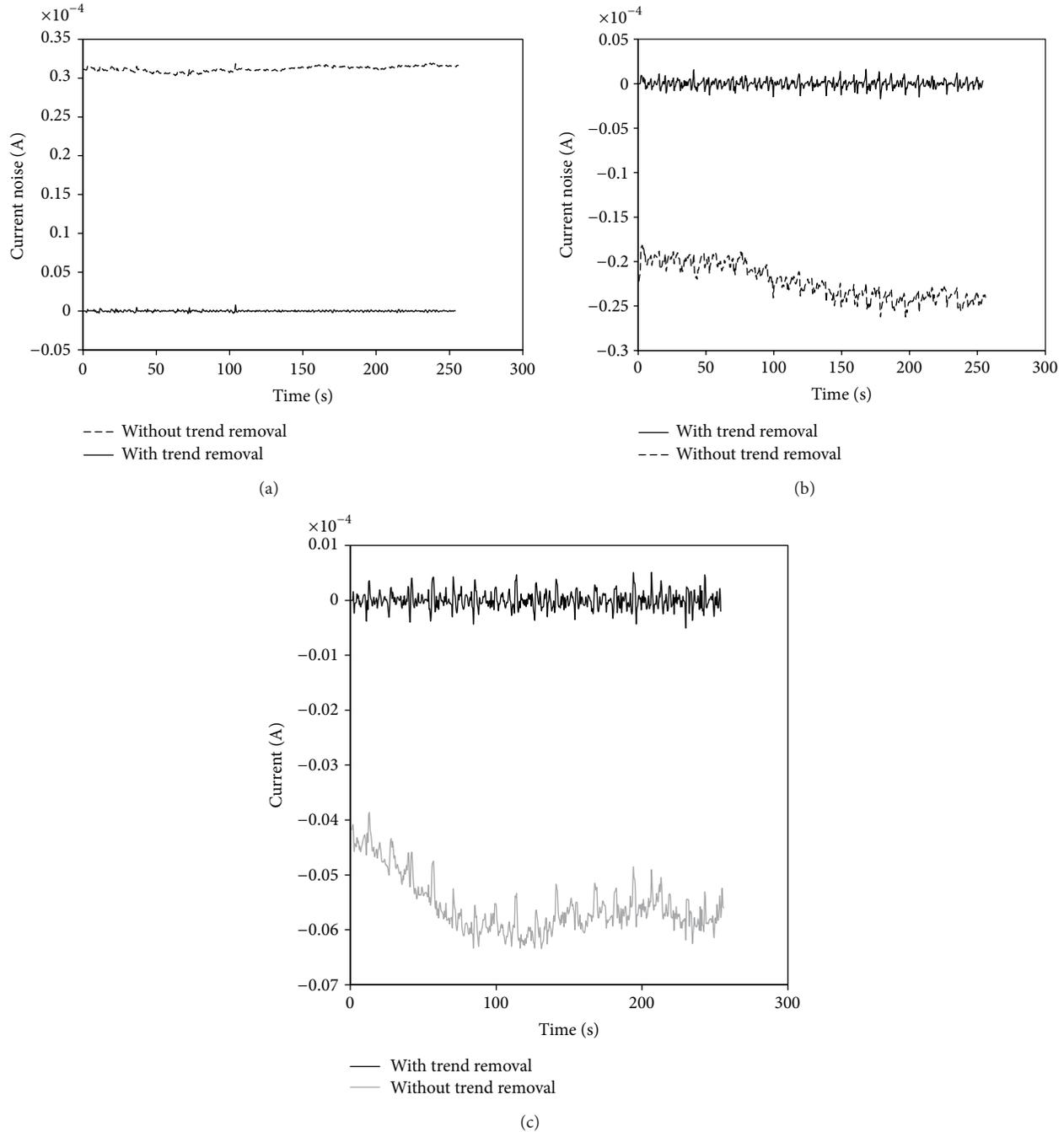


FIGURE 5: Electrochemical current noise diagrams before and after trend removal for composite coatings obtained from plating bath containing different concentration of nano- Al_2O_3 particles: (a) blank, (b) 5 ppm, and (c) 20 ppm.

dissolution of nickel occurs at open circuit potential, leading to the enrichment of phosphorus on the surface layer [6, 35, 36]. The enriched phosphorus surface reacts with water to form a layer of adsorbed hypophosphite anions (H_2PO_2^-). This layer in turn will block the water supply to the electrode surface, thereby preventing the hydration of nickel, which is considered to be the first step to form either soluble Ni^{2+} species or a passive nickel film [36, 37].

3.4. Scanning Electron Microscopy Analysis. Figure 6 shows SEM images of Ni-Cu-P- Al_2O_3 nanocomposite containing different concentrations of nanoparticles. It is obvious that nano- Al_2O_3 particles assembled to form the agglomerates and are embedded in the Ni-Cu-P matrix through the codeposition process and thus affect some coatings properties [18]. Figure 6(b) illustrates some nodular structures of these nanoparticles that were uniformly dispersed in coating

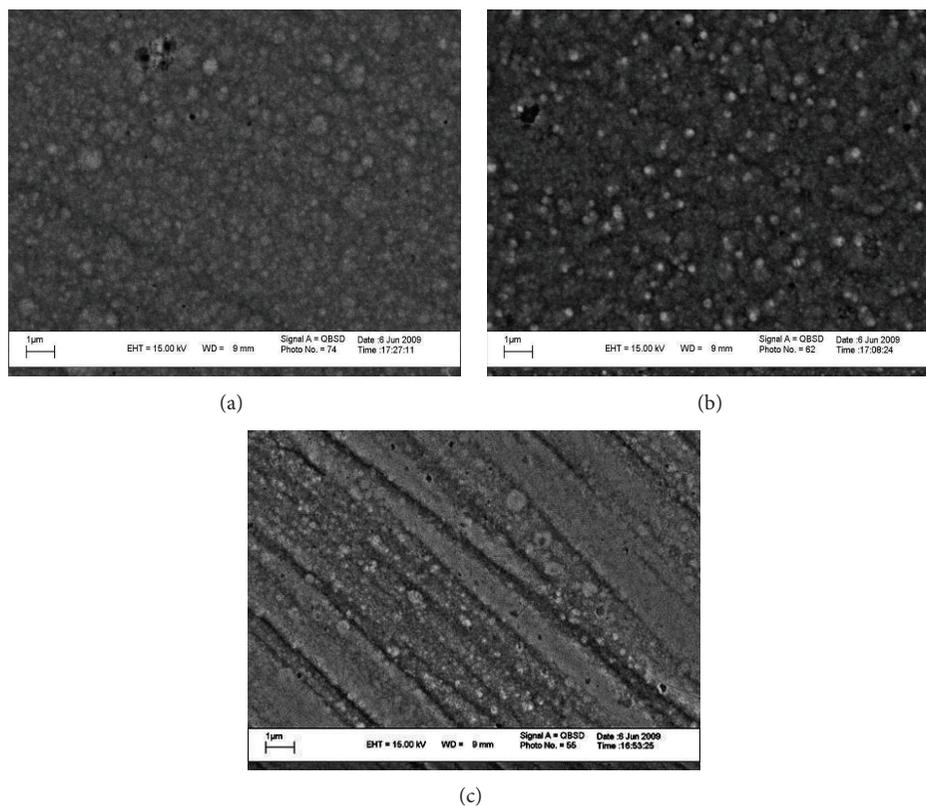


FIGURE 6: SEM images of Ni-Cu-P- Al_2O_3 electroless composite coatings obtained from plating bath containing different concentration of nano- Al_2O_3 particles: (a) 5 ppm, (b) 20 ppm, and (c) 1000 ppm.

surface with respect to coatings obtained from plating baths containing other nanoparticle concentrations. According to these images, with an increase in nanoparticles concentration above 20 ppm in the bath, the nanoparticles content in the coating surface has decreased. Based on these observations, it may be said why the coating plated from bath containing 20 ppm nano- Al_2O_3 particles shows maximum corrosion resistance which has been confirmed by electrochemical methods.

4. Conclusion

In this study Ni-Cu-P and Ni-Cu-P/nano- Al_2O_3 composite coatings were prepared by electroless deposition. The presence of nano- Al_2O_3 particles in the coatings has an effective inhibitive action on corrosion performance of Ni-Cu-P composite coatings on mild steel in 0.5 M H_2SO_4 solution. The results obtained from EIS and polarization measurements show that the addition of alumina nanoparticles in the electroless bath enhanced the surface corrosion resistance. The highest corrosion resistance was obtained at the nano- Al_2O_3 particles concentration of 20 ppm in the plating bath. The values of noise resistance after trend removal were in good agreement with charge transfer resistance and polarization resistance obtained from other electrochemical measurements. SEM images proved that nano- Al_2O_3 particles assemble to form the agglomerates and were embedded in

the Ni-Cu-P matrix and were dispersed uniformly in the coating surface.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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