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Corrosion Behaviour and Morphological Analysis of Ni/Cu Nanolayer Coating in Salt Solution

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Abstract: *The electrochemical corrosion studies of Nickel/Copper (Ni/Cu) compositionally modulated multilayer nanolayer (CMM) in 3.5 wt% of Sodium Chloride (NaCl) solution at room temperature were investigated using potentiodynamic polarization (PDP) method. A multi-nanolayer of Ni/Cu with the total thicknesses of 3 μm was successfully produced on Cu substrate via electrodeposition process through dual bath technique (DBT). The electrodeposition with 3 different sublayer thicknesses (40 nm, 80 nm and 100 nm) was produced by varying the deposition time. The results of electrochemical experiment indicate that Ni/Cu multi-nanolayer coating have superior corrosion resistance in 3.5 wt % of NaCl solution than the uncoated Cu substrate. The corrosion resistance is increased when the sublayer thicknesses decrease. The morphological analysis of Ni/Cumulti-nanolayer after corrosion testing was examined. The results shows that the uncoated Cu substrate corrode faster if compared to that of Cu substrate coated with Ni/Cu multi-nanolayer coating.*

Keywords: *Multilayer, Cu/Ni, Corrosion Resistance*

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1 Introduction

Corrosion is a degradation attack on metal's properties by chemical (dry corrosion) or electrochemical (wet corrosion) process due to the interaction of that particular metal with its environment [1-2]. Controlling corrosion of this metal via metal surfaces protection which can act as a barrier to offer sacrificial protection, inhibit corrosion and some of the methods using treatment on the surface of a metal in order to increase its resistance to corrosion [3]. Recently, composite modulated multilayer (CMM) has received attention among researchers because this

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multilayer presents improved properties compared to the corresponding pure metals. It has been intensively studied for their attractive properties such as electrical, mechanical, optical or magnetic properties [4-5].

Moreover, most of nanomaterials produced by mechanical method is believed to have lots of microstrains and pores at their grain boundary regions which are particularly susceptible to corrosion. These microstrains and pores could be the initial sites for the corrosion attack [5-6]. In this research work, the electrodeposition method has been chosen to fabricate a multi-nanolayer coating on Cu substrate because it involves low cost and ease of operation [7]. The nanolayer produced by electrodeposition is expected to produce structures which are dense and no pore [5-6].

Electrodeposition of CMM Ni/Cu can be fabricated by two techniques either by single bath technique (SBT) or dual bath technique (DBT). Dual bath technique offers several advantages as compared with single bath technique. It effortlessly controls the production of multilayer structure because there is no limitation in the production of layered structure and it is an easily prepared operation in producing coating with several hundreds of thicknesses [7-8]. In this present research, CMM Ni/Cu having sublayer thicknesses of 40 nm, 80 nm and 100 nm have been deposited. The evaluations of corrosion behaviour of multi-nanolayer deposits based on their electrochemical activities were tested using potentiodynamic polarization method. After 24 hour of corrosion test the samples were analysed by optical microscope. Next, the effect of multilayer system on the corrosion resistance was discussed.

2 Experimental

The electroplating solutions were prepared from analytical reagent composed of 300 gL^{-1} nickel sulphate ($NiSO_4.6H_2O$), 50 gL^{-1} nickel dichloride hexahydrate ($NiCl_2.6H_2O$) and 40 gL^{-1} boric acid (H_3BO_3) which were dissolved in deionized water [9]. Electrodeposition process was carried out on Cu substrate, at pH 4 and temperature of 23 $^{\circ}C$. A direct current (DC) power supply was used to provide consistent current during the electrodeposition process. The production of different sublayer thicknesses of the coating follows Faraday's Law by using different deposition time in order to produce certain thicknesses. The time measurement was measured using a digital watch. A solution of 1000 ppm of sodium chloride (NaCl) was prepared by dissolving 35 g of NaCl in distilled water. From this stock solution, 3.5 wt% of NaCl was used for the corrosion test. All samples with different sublayer thicknesses (40 nm, 80 nm and 100 nm) were tested.

2.1 Electrochemical studies

DC corrosion test was carried out with the aid of Potentiostat/Galvanostat Model WPG 1000 device. The electrochemical set up consists of platinum electrode as counter electrode, Ag/AgCl/1.0 M KCl electrode as reference electrode and the sample as working electrode. The working area of test samples with dimension of 3.5 cm x 4 cm x 1 cm was used with exposing area of 14 cm^2. The samples were gradually polished with silicon carbide (SiC) paper up to 2000 grid. Then, all samples were micro polished with alumina powder to produce mirror finish surface. Subsequently, all samples were degreased in ethanol and ultrasonically cleaned.

Prior to the polarization measurements, all samples were immersed in the solution for about 60 min. The corrosion rate, corrosion potential E_{corr}, and corrosion current I_{corr} were determined via PDP method.

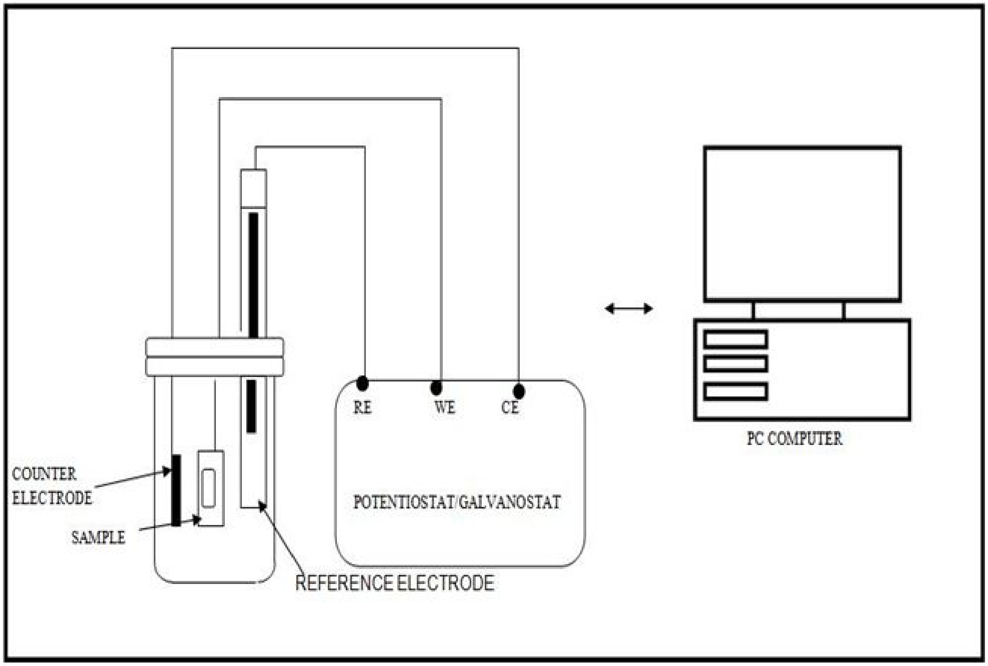


FIGURE 1. A schematic representation of corrosion test for Ni/Cu multi-nanolayer by potentiodynamic polarization method

3 Results and Discussion

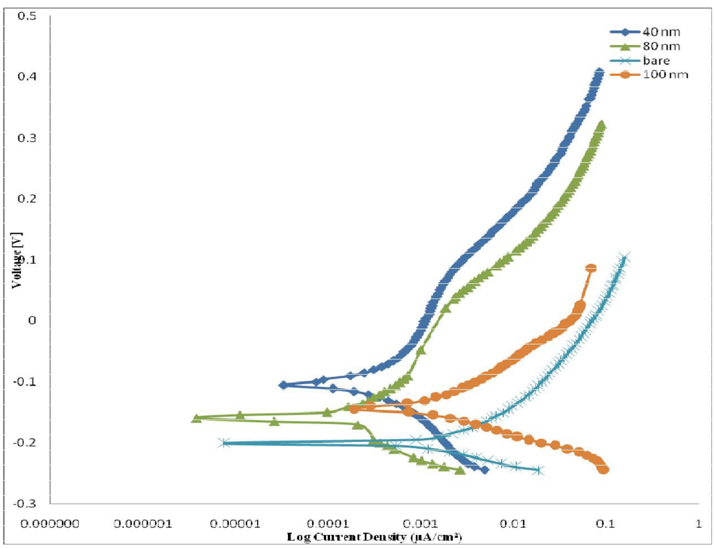


FIGURE 2. Representative potentiodynamic polarisation curves of Ni/Cu multilayer and Substrate

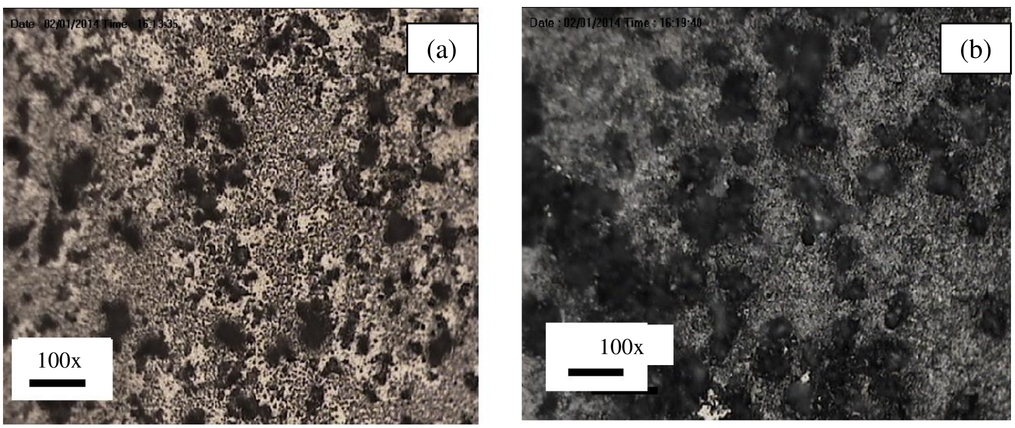
Potentiodynamic polarization curves of CMM Ni/Cu coating with different number of sublayers and uncoated Cu substrate in 3.5 wt. % NaCl solutions at room temperature are shown in Figure 2. NaCl has been chosen as a medium for corrosion test solution because of Cu/Ni alloy or multilayer is commonly used in application of sea water [4]. The measured of E_{corr}, and other electrochemical parameters for Ni/Cu multilayer are tabulated in Table 1. The shape of overall polarization curves of the coating and substrate obtained in NaCl medium is similar to those found by other researcher [5].

The corrosion resistance of Ni/Cu multi-nanolayer is significantly improved when whole polarization curve was shifted towards the region of higher potential. The values corrosion potential E_{corr} are -166.49 mV and -104.46 mV for 40 nm and 100 nm of Ni/Cu multi-nanolayer, respectively, whereas the Cu substrate shows corrosion potential value of -172.42 mV which indicate that the Cu/Ni multi-nanolayer exhibit good corrosion resistance compared to Cu substrate. The corrosion current density I_{corr} of 40 nm is 541. 48 µA/cm² and the Cu substrate are 1.035 mA /cm². It could be seen that the corrosion current density of the Ni/Cu multi-nanolayer was found lower than that of the Cu substrate. The lower corrosion current density I_{corr} value indicates that the material resist to the corrosion attack. Moreover, from the corrosion measurements the corrosion rates of Ni/Cu multi-nanolayers are smaller than the corrosion rate of Cu substrate. Ni/Cu multi-nanolayer with sublayer thicknesses of 40 nm shows the best corrosion rate which is 8.9 (10^2 mm/year) lower than the Cu substrate as tabulated in Table 1.

When at smaller sublayer thickness, the number of layer will be increased in order to achieve the total thicknesses 3 µm. Due to that reason, as the numbers of interface increases, more numbers of micro-pores are blocked. This condition will improve the corrosion resistance of the multilayer coatings [10]. Electrodeposition is known to provide dense and pore-free nanostructured metals, which make them less prone to certain types of corrosion such as pitting [5].

Table 1: Electrochemical parameter of CMM Ni/Cu coating and uncoated Cu substrate in 3.5 wt. % NaCl

Sublayer Thicknesses	E_{corr} (mV)	CR (10^{-2} mm/yr)
Cu Substrate	-172.42	86.15
100 nm	-166.49	37.51
80 nm	-162.31	9.91
40 nm	-104.46	8.39



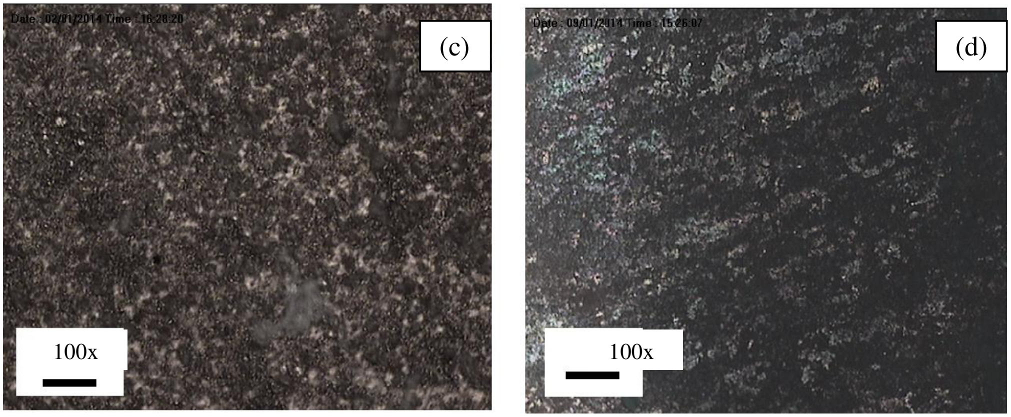


FIGURE 3: Optical microscope of (a) 40 nm (b) 80 nm (c) 100 nm and (4) substrate in 3.5 wt. % NaCl solutions.

Figure 3 shows the corroded surface morphology of multi-nanolayer with sublayer thicknesses of 40 nm, 80 nm, 100 nm and uncoated Cu substrate in 3.5 % NaCl. After 24 hours of corrosion testing, the morphology of the corroded samples was observed with an optical microscope. From the observation, Ni/Cu with sublayer thicknesses of 40 nm and 80 nm shows that there is only the present of nickel oxide deforms on the surface. Moreover, Ni/Cu multi-nanolayer of 100 nm shows a numerous of pits and the portions of specimen corroded were distributed to all the surface area whereas, the Cu substrate showed homogeneous corrosion with numerous small pits, few large and deep pits. This result correlates with the result findings in potentiodynamic test.

4 Conclusion

Ni/Cu multi-nanolayer coatings were electrodeposited on Cu substrate. The corroded surface morphology of the coating shows that the CMM Ni/Cu coating has better corrosion rate than uncoated Cu substrate. The results obtained from corrosion potential measurement for CMM Ni/Cu coating shift to more positive potential value than Cu substrate. Based on results from electrochemical measurement, the current density of uncoated Cu substrate with higher corrosion current density will indicates that the uncoated Cu substrate prone to corrosion attack rather than CMM Ni/Cu coating. The corrosion resistance is increased when the sublayer thicknesses decrease.

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