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Study of Thin Film Copper Electrodeposition on Carbon Substrate for Thin Film Battery Electrode Application

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Abstract: The electrodeposition of copper onto carbon substrate was studied in 0.5 M of $CuSO_4$ solution at various applied voltages; 2.0, 4.8 and 6.0 V. The electrodeposition was carried in an electrochemical cell with copper as the anode and carbon as the cathode. The influence of electrodeposition parameters on the thickness of deposits and surface roughness of copper films were studied in detail using Atomic Force Microscopy (AFM). The current value increases with the increasing of applied voltage. Charge-discharge test was performed in 0.5 M and 1.0 M of HCl, and revealed that high concentration of electrolyte resulted high surface roughness and thickness of copper film.

Keywords: Electrodeposition, Copper, Thin film, Surface roughness, Film Thickness.

1.0 Introduction

Electrodeposition becomes an important method for coating process in industry for many types of applications. In general, electrodeposition yields good adherence of coatings on the surface of substrate. Electrodeposition process has many advantages, for example: it requires low-cost than other techniques such as plasma vapour deposition (PVD) and chemical vapour deposition (CVD), effective in coating surface where able to coat even on a difficult surface and able to control the thickness and composition of film by adjusting the parameters [1]-[2].

Copper is mainly used in coating industry due to its ability to cover the minor defects in the base metal. Besides that, copper has advantages of high plating efficiency, excellent coverage even on gap filling means that a difficult-to-plate parts and highly conductive. These advantages make the copper become more importance in the coating process including coating for printed wiring boards or steel wire used to conduct electricity [3].

Thin film batteries are the worldwide high-technology demand. Many material researchers in various fields are making researches to develop new materials with better fabrication methods for the thin film batteries application [4]. Thin film is a two dimensional layer of material where the thickness ranging from nanometer to several micrometers [5]. Thin film electrode can also be fabricated through irradiation of polymer, such as polystyrene to become carbanacous materials [6] and recently polystyrene bormbarded by pulse ultra violet laser irradiation have been found to emit white light [7]. These researches can be used to assist in fabricating thin film battery and light emmiting diode (LED).

This paper presents the study of thin film copper electrodeposition on carbon substrate for thin film battery application.

2.0 Methodology

Anode and Cathode:
Copper rod (99.90% purity, diameter 3 mm, length 1 m) and carbon fiber epoxy sheet (length 300 mm, width 300 mm, thickness 1 mm) were purchased from RS Components, Selangor. Copper and Carbon fiber samples samples were cut into 1.0 cm × 1.0 cm area and cleaned using distilled water and acetone/ethanol, later dried at room temperature.

Electrolyte:
0.5 M of $CuSO_4$ solution were prepared by dissolving 124.84 g of $CuSO_4$ powder in 1 L of distilled water.

Electrodeposition:
The copper electrodeposition was performed in 50 mL of $CuSO_4$ solution with copper as anode and carbon as cathode. The distance between

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electrodes is 2 cm . The applied voltage was varied with 2.0, 4.8 and 6.0 V.

Charging-discharging Battery:
Discharging test of the battery were conducted by connecting with multimeter to measure the voltages. Charging test were performed by applying the average voltage and monitoring the currents via multimeter. Both charging and discharging test were conducted for 1 hour.

Characterization:
The deposited copper films on carbon substrate were observed using AFM (Hitachi 5100N) in non-contact mode technique. The Gwyddion software was used to analyse the surface roughness and film thickness of the images obtained over 5 µm × 5 µm.

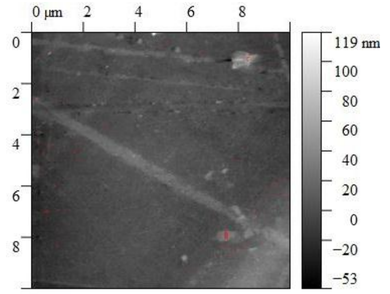
3.0 Results and Discussion

Table 1 shows the values of current flow copper during the process of electrodeposition. It can be seen from Table 1 that the highest current resulted from higher deposition voltage. High applied voltage causes the electrode tobe released, thus provide high current.

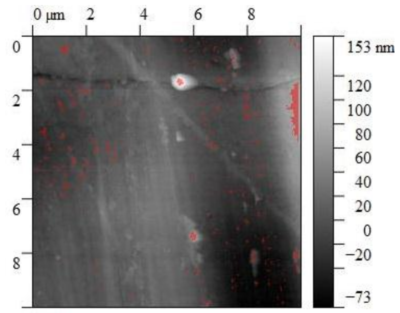
Table 1 Results from electrodeposition experiments using various potentials.

Sample	Deposition voltage (V)	Current (A)
1	2.0	0.09
2	4.8	0.19
3	6.0	0.38

Figure 1 presents the SEM images of copper thin films. In Figure 1(a) the copper film is 68 nm thick, in Figure 1(b) the copper film is 118 nm thick and in Figure 1(c) the copper film is 338 nm thick. The red marks indicated the grains growth on the surface of copper film. The fined sized copper grains on the background of film are observed in Figure 1(a). It shows that, at 2.0 V, only small amount of copper had deposited onto carbon substrate. In Figure 1(b), the formation of copper grains were increased, which caused by an additional applied voltage during electrodeposition. For higher voltage, at 6.0 V, the aggregation of grain growth had increase the coverage of deposits.



(a)



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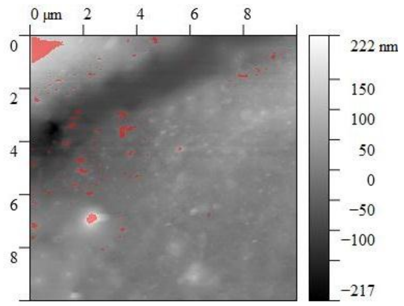
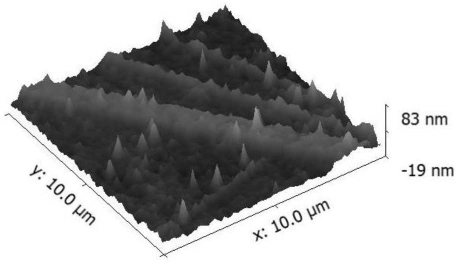


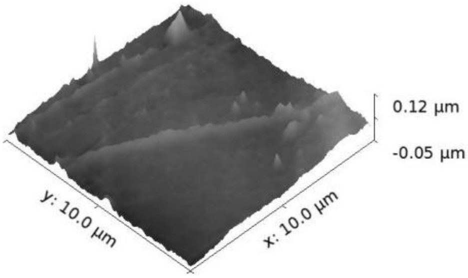
(c)

Figure 1 Surface morphologies of copper thin films on carbon substrate at (a) 2.0 V, (b) 4.8 V and (c) 6.0 V.

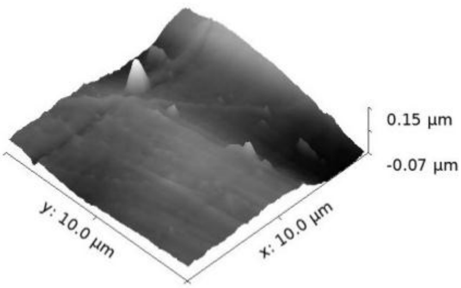
Figure 2 shows the 3-dimensional images of copper films deposited on carbon at potential 2.0, 4.8 and 6.0 V. As can be seen in Figure 2, the film thickness increased with the increasing of applied voltage. At 2.0 V, in Figure 2(b), the grain formation was less than at 6.0 V, as can be seen in Figure 2(d). At low potential, the coating was not good compared to coating at high potential. Thus, coating at high potential can increase the film thickness.



(a)



(b)



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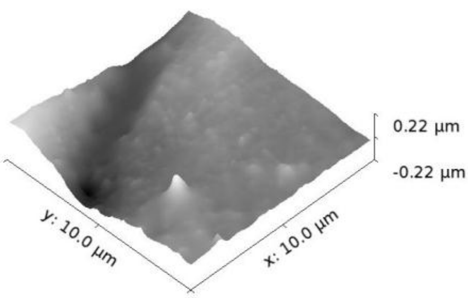

(d)

Figure 2 3-Dimensional AFM images of (a) bare carbon and deposited copper on carbon substrate at various potentials (a) 2.0 V (b) 4.8 V and (c) 6.0 V.

Table 2 shows the complete analysis of surface roughness and film thickness. The highest applied voltage (Sample 1) brings highest surface roughness and film thickness. At higher voltage, the deposition is high leading to increase in grain growth, thus increase the surface roughness. Sample 1 results the lowest value of surface roughness RMS and film thickness due to low potential that applied during electrodeposition.

Table 2 Surface roughness RMS and film thickness resulted from AFM analysis.

Sample	**Surface roughness RMS (nm)**	**Thickness measured (nm)**
1	11.1	68
2	26.9	118
3	48.3	338

Charge-discharge test were performed in two different electrolyte concentrations; 0.5 M and 1.0 M of HCl for duration of 60 min. Table 3 summarizes the values of voltage and current obtained from charge-discharge test. As can be seen from Table 3, the potential values obtained from discharge test are constant and the values increased to 0.3 V during Sample 3(b). During charging, at concentration 0.5 M HCl, the currents were increasing with the increases of applied voltage. At Sample 3(b), the current had decreased to 0.15 A. Meanwhile, for the concentration 1.0 M HCl, the currents were decreased with the increasing of applied voltage. Some errors occurred during charging and discharging process, leading to incorrect values. The errors could be the layer resistance that caused by the previous deposited layer.

Table 3 Values for voltages and currents obtained from charge-discharge experiments.

Sample	**HCl concentration (M)**	**Potential (V)**	**Current (A)**
1(a)	0.5	0.1	0.16
1(b)	1.0	0.1	0.22
2(a)	0.5	0.1	0.34
2(b)	1.0	0.1	0.16
3(a)	0.5	0.1	0.15
3(b)	1.0	0.3	0.15

4.0 Conclusion

Copper was successfully deposited onto carbon substrate via electrochemical deposition process in $CuSO_4$ solution. Electrodeposition of copper at high applied voltage produces high current value. AFM analysis revealed that high surface roughness and thickness of copper film resulted from high applied voltage during electrodeposition process. Characterization of battery charge-discharge showed that the electrolyte concentration affects the grain growth on film. High concentration of HCl solution increase the surface roughness and film thickness.

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