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Deposition Time Variation on Thickness and Resistivity of Cu/Ni Thin Film Obtained by Magnetic Field-Assisted Electroplating Process

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Abstract. Thin films of Cu/Ni have been synthesized by the electroplating method assisted by a magnetic field on the variation of deposition time. The use of magnetic fields in the deposition process to increases the rate of electroplating and reducing the effect of hydrogen evolution so that the surface morphology of the layers becomes homogeneous. Deposition time affects the thickness of the formed layer. The electroplating is carried out at a DC voltage of 1.5 volts, the magnetic field of 200 gauss, a solution temperature of 60 ºC, and an electrode distance of 4 cm with Cu as a cathode and Ni as an anode. The electrolyte solution made from the mixture of H_3BO_3 (30 g), $NiCl_2$ (195 g), $NiSO_4$ (45 g), and H_2O (750 ml) and deposition times varied from 5 s - 45 s with intervals of 10 s. The characterization was done by thickness test using the calculation of the mass, and resistivity of the Ni layer by using the four-point probe. The results indicate that the deposition time is proportional to the thickness of the formed layer and inversely proportional to the resistivity. Ni layer thickness ranges from 1.48 $\times 10^{-2}$ µm to 5.02 $\times 10^{-7}$ µm. while the sheet resistivity is from $(4.87 \pm 0.02) \times 10^{-3}$ Ω/sq to $(1.38 \pm 0.06) \times 10^{-3}$ Ω/sq.

1. Introduction

These Technological developments continue to increase to be more effective and accurate as a basis for conducting research related to innovation in the field of technology. One of the techniques developed in the research is sensor technology. The use of sensors is found in almost all fields [1,2], including in the field of biology related to embryo and gamete preservations, preservation of organ transplants, and preservation of blood products, and preservation of food [3,4].

Preservation usually uses a medium with low temperature such as liquid nitrogen that has a minimum temperature of 200 ºC. Studies on materials that have the potential to be used as low-temperature sensors are continuously carried out [5]. Materials that are sensitive to low temperatures can be made from a thin film by utilizing changes in the resistance when the temperature changes. This material is a type of RTD (Resistance Temperature Detector) [6].

Cu and Ni and these combinations in the form of alloy CuNi and thin film Cu/Ni is a material that has the potential to be used as a low-temperature sensor [7-10]. Cu/Ni is a kind of temperature sensor that works based on a Resistance Temperature Detector (RTD). Therefore study about the resistance of the elements in responding to the temperature is an important thing. As sensor material, Cu, Ni or Cu/Ni film are elements that have excellent physical properties that are a linear relationship between resistance and temperature [9]. Plating Ni material on Cu substrate will increase the resistivity of Cu/Ni.

In this research, we synthesize Cu/Ni film by electroplating on the various time depositions. The time deposition in plating will influence the thickness of the film, and also the thickness will influence the resistivity. For metal, resistivity is inversely proportional to the thickness. The more thickness getting less resistivity. It is whished that the high resistivity of Cu/Ni will have a correlation to the increasing sensitivity [5,11]. The problem is the surface morphology of Ni film that is not uniform, and still contains vacancies. By that condition, the resistance of sample in different location become not homogen.

To improve the microstructure on the surface of the sample in this study used electroplating assisted by magnetic fields with the direction perpendicular to the electric field between anode-cathode (E). The utilization of magnetic field (B) in that direction will generate the Lorentz force on the direction perpendicular to E and B. Interaction between the Lorentz force and Ni ions in the electrolyte will generate the magnetohydrodynamic (MHD) effect [12,13]. MHD will impact to arising the mass transport of deposits [14,15]. This is can be seen from the increasing the current along the deposition process [16,17]. Beside that MHD will make the film morphology, in this case, Ni film be homogeneous [12,18], the effect of hydrogen evolution reactions be reduced [13,19].

2. Research Methods

2.1. Material research

The The material used in this research is copper (Cu) plate as a substrate with the size of 2.5 cm × 1.0 cm × 0.02 cm and nickel (Ni) with the size of 3 cm × 1.0 cm × 0.4 cm as a coated material. The electrolyte makes from a mixture of nickel sulfate ($NiSO_4$), nickel chloride ($NiCl_2$), boric acid (H_3BO_3), and distilled water of 195g, 45g, 30g, 750ml, respectively. Alcohol 96% is used to rinse the substrate. Toothpaste is used to clean the substrate of impurities and grease sticking, and metal polisher is used to polish the substrate.

The equipment used for preparing the sample consisted of 1000 ml beaker glass used as a plating bath; Magnetic stirrer is used to stir the solution; the electric stove is used to heat the electrolyte solution. Vernier thermocouple is used to control the temperature of the solution stable at 60 °C, the DC voltage set on 1.5 volts as a power supply. PA-214 Ohaus scale with accuracy to 10-4 g was used to measure the mass of Cu substrate before plating and Cu/Ni film after plating. An ultrasonic cleaner is used to clean the sample and two multimasters that set as voltmeter and amperemeter were used to measure the voltage and current in for point probe with accuracy to 10^{-3} mV and 10^{-2} mA respectively. The voltage and current data will be used to determine the sheet resistivity of the Cu/Ni film.

2.2. Method of experiment

In At the preparation stage, the cut copper substrate was polished with autosol then cleaned with toothpaste and rinsed with distilled water at 60 °C gradually until three times. The last stage substrate was rinsed using 96% alcohol in an ultrasonic cleaner for 3 minutes. The sample then hairdryed. After drying the sample was wrapped in tissue, and then inserted in a plastic clip and strored in drybox. Electrolyte solutions are stirred in a measuring cup using a magnetic stirrer for 4 hours.

The next stage is the electroplating process. An electrolyte solution is heated to 60 °C. Setting the electrolyte temperature 60 °C will produce a more flexible layer [20]. Then poured in plat bath — the tool design as in Fig. 1 the magnetic field to be set at 200 gauss. Nickel anodes and copper cathodes were connected to a 1.5 volt DC voltage. Data was acquised in the form of deposition time, current, and voltage with smpling rate 20 data/s. Deposition time was varied from 5s-45s with intervals of 5 s.

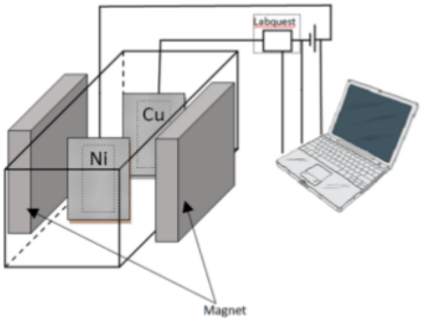

Figure 1. Schematic diagram of the experimental setup

2.3. *Method of experiment*

The Ni layer thickness was obtained through measuring the mass difference in Cu substrate and after deposition with Ni by the Lowenheim equation,

$$\delta = \frac{m_2 - m_1}{\rho A} \tag{1}$$

And

$$S_\delta = \sqrt{\left(\frac{\partial \delta}{\partial m} S_m\right)^2 + \left(\frac{\partial \delta}{\partial p} S_p\right)^2 + \left(\frac{\partial \delta}{\partial A} S_A\right)^2} \tag{2}$$

Where δ is sheet thickness, m_1 and m_2 is mass of Cu substrate and Cu/Ni sample respectively, ρ is density, A is cross-section area, S_δ is the error of thickness. The sheet resistivity was determined based on a formula,

$$R_s = \frac{\pi}{\ln 2} \frac{V}{I} \tag{3}$$

If the measurement is done by varying I and recording the value V, then by linearization of data (V_i, I_i) we found a as a slope of the curve, and the resistivity becomes,

$$R_S = \frac{\pi}{\ln 2} a \tag{4}$$

The error of R_s is,

$$S_{R_S} = \frac{\pi}{\ln 2} S_a \tag{5}$$

where,

$$S_a = S_{\hat{y}} \sqrt{\frac{N}{N \Sigma x_i^2 - \left(\Sigma x_i\right)^2}} \tag{6}$$

and,

$$S_{\hat{y}} = \sqrt{\frac{\Sigma\left(y_i - \hat{y}\right)^2}{N-2}} \tag{7}$$

with N is the amount of data.

3. Result and Discussion

3.1. Current in the deposition process

In figure 2 a deposition current is displayed during the electroplating. From this picture, it appears that all of the current used in a deposition is relatively stable at around 0.45 A. Unfortunately the time for start stable is different. When the plating process starts the current from zero then rises. For deposition at 15s, 25s, and 35s, currents fast to reach stability in contrast to deposition at 5s and 45s that appear to be rather slow to achieve a stable current of about 0.3s. With different initial stable conditions, this will affect the deposit product attached to the Cu substrate at the cathode. The presence of the ripple, especially in a deposition for 35s, describes the number of electrons passing through the amperemeter. This also illustrates the amount of Ni ions leading to the cathode.

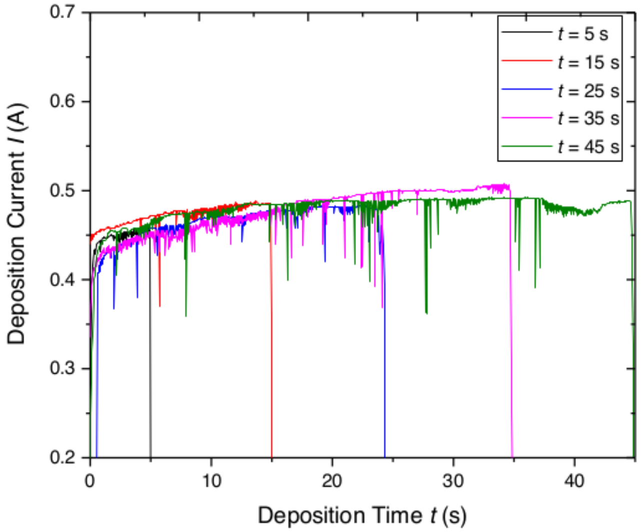

Figure 2. Deposition current at various deposition time

3.2. Film Thickness

The results of measuring the thickness of the film from various variations t with a magnetic field of 200 gauss are shown in Figure 2. It appears that the thickness of the layer is directly proportional to the time of deposition according to the equation.

$$\delta = 0.01195t - 0.05384 \tag{8}$$

where δ is the thickness and t is deposition time. Determination index of this graph is 99%, that shows the presence of a strong relation between time and the thickness. The highest thickness values obtained at 45 s deposition time are 50,2 µm, while at the deposition time 5 s the smallest film is 1,48 µm.

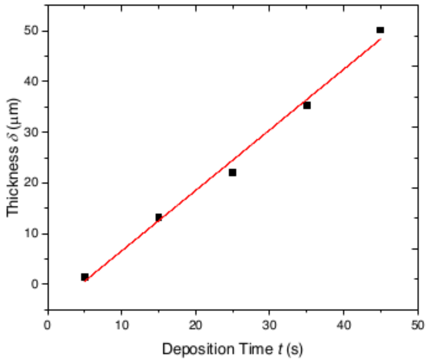

Figure 3. Layer thickness of the Ni layer at various deposition time

In accordance with Faraday's Law that the mass formed at the electrode is proportional to the equivalent weight, current strength, and deposition time.

$$M = \frac{e\,i\,t}{F} \tag{9}$$

where M the mass of the Ni deposit, and e is the equal weight, i is the current in the deposition process, F is the Faraday constant. Other researchers [20] also obtained the same results that the longer the deposition time, the thicker the layer formed.

As a comparison, we also deposited Ni on Cu substrate during 5s without applying the magnetic field. The thickness of Ni is 2.9 µm. This result is contrary to the purpose of using a magnetic field which is to increase the mass transport of Ni ions to the cathode. The fact that deposit at the cathode is less than the deposit obtained without magnetic fields. The thickness of the deposit only exceeds the deposit without B when the deposition time is 35s. It is assumed that the use of a magnetic field of 200 gauss generates a Lorentz force which causes some Ni ions to deviate from the target cathode, by taking into account that the electrode distance of 4 cm is a significant distance to deviate the Ni ions. Another possibility is that the activation of the magnetic field precedes the activation of the electric field so that Ni atoms are attracted to the cathode but not strong. When field B is removed the Ni does not stick. When the electric field is activated, the flow of Ni ions close to the substrate is blocked with nickel particles that have been attached to the cathode. Therefore, only a little Ni deposit is formed. To avoid that, activation of the magnetic field and electroplating process needs to be carried out together.

3.3. Formatting author affiliations

In Figure 2 is shown the sheet resistivity of Cu and Cu/Ni. From the figure, it appears that the sheet resistivity of Cu increases after being deposited with Ni. In line with the increase in deposition time, the sheet resistivity of Cu/Ni become decrease. The curvature of the curves for R_s-Cu and R_s-Cu/Ni shows that the five Cu substrate used has different R_s. This affects the value of R_s-Cu/Ni. But from the increase in Cu/Ni resistivity from Cu resistivity has a linear relationship to the deposition time according to the equation.

$$\Delta R_s = -4.8\text{E-}07t + 0.0013\ (\Omega/\text{sq}) \tag{10}$$

Where the determination index $R^2 = 0.99$. The smallest ΔR_s is 1.25×10^{-3} Ω /sq that corresponds to 5s deposition time and the highest ΔR_s is 1.23×10^{-3} Ω/sq related to deposition time 45s.

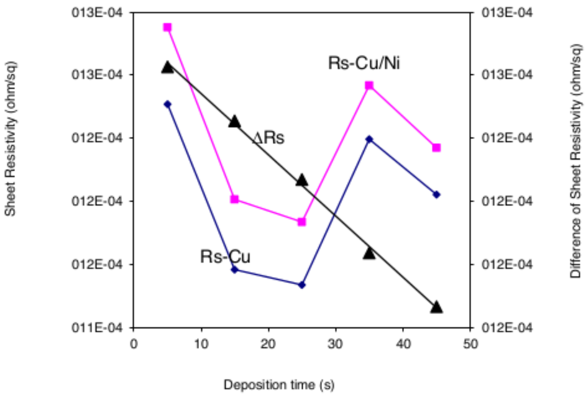

Figure 4. The sheet resistivity of Cu and Cu/Ni

6 Conclusion

Based on the results of the study, it was found that the Ni layer on the Cu substrate had succeeded to be synthesized in the variation of the electrolyte solution temperature. In this study, it is also known that the thickness of the Ni layer is proportional to the temperature of the electrolyte solution and inversely proportional to its resistivity.

Acknowledgments

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