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Thickness and Resistivities of Cu/Ni Film Resulted by Electroplating on the Various Electrolyte Temperature

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Abstract. Synthesis of Cu/Ni thin films has been carried out by electroplating method assisted by magnetic fields at variations in the temperature of the solution. The use of magnetic fields in the deposition process is to accelerate mass transport, reduce the reaction effect of hydrogen evolution, improve surface morphology. An increase in the temperature of the solution can accelerate the rate of Ni ions from the anode to the cathode. The electroplating process is carried out at a DC voltage of 1.5 volts, a magnetic field of 200 gauss that is perpendicular to the electric field, within 5 seconds, and the electrode distance is 4 cm. The solution temperature varied from 40ºC ─ 80ºC at intervals of 10 ºC. Characterization is done by testing the thickness and resistivity of the Ni layer. Thickness test is done with the help of calculations from the results of weighing Ni. The test of sheet resistivity is done by means of a 4 point probe

The results showed that the greater the temperature of the solution the thicker the Ni layer. The thickness of the Ni layer ranges from (0.09 ± 0.001) µm to (0.38 ± 0.006) µm which is the opposite to the resistivity of the chip, namely the higher the temperature of the solution the smaller the sheet resistivity. The size of the sheet resistivity is from (1.31 ± 0.02) Ω/sq. To (1.38 ± 0.06) Ω/sq to identify relevant articles in literature searches, great care should be taken in constructing both

1. Introduction

The progress of research in the field of material physics is in line with the times. Some materials are considered to have advantages such as easy to get, resistant to corrosion, resistant to wear, have strength, hardness, and low cost.

Thin layers are increasingly needed in various fields such as in industry, health, education, security and in other fields [1-3]. The thin layer is a layer of metal whose thickness ranges from the order of angstroms to microns [4,5]. In the medical field to preserve vaccines so that they remain stable and can last for a long time, preservation is carried out at low temperatures, namely cryogenic freezing below -150ºC [6]. Similarly, cryonic methods have been developed to preserve organs so that for a long time the tissue is still alive and can be used at any time needed.

At this temperature, objects are preserved on the freezing unit. For that, we need a thermometer that can measure these low temperatures. One material that can be used as a low-temperature sensor is a combination of Cu and Ni so CuNi or Cu/Ni layers are produced

One of the benefits of Ni, when combined with Cu, is to increase the resistivity. Resistivity is one parameter that play an important role in determining sensitivity. The higher the resistivity of the material the more sensitive the material, because of the greater the resistivity range available to hold

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the temperature. This is because the material has a greater resistivity range to accommodate the temperature changes.

Ni has a higher resistivity (7.3 μΩcm) than Cu (1.7 μΩcm) [7] so that the combination Ni with Cu will increase the resistivity of Cu. In the study of metal, the Cu and Ni are two materials that is not difficult to combine because they are both transition metals that have almost the same radius of the atom, which is 1.25Å for Ni and 1.28Å for Cu so that if Ni is coated with Cu both of them will produce a good microstructure [8].

Cu and Ni did not difficult to be combined because both included transition metals that have almost the same atomic radius, namely 1.25Å for Ni and 1.28Å for Cu. If Ni is coated to Cu, a good microstructure will be produced [8], that is a parameter to produce advantage material properties.

One of the methods to make the thin film is electroplating [9] is the process of metal deposition over other metals using electrolysis [10]. Giving direct current to the solution causes a reduction process in the cathode and oxidation at the anode. In the process of electroplating, the temperature and magnetic field are factors that can affect the quality of the coating. The increase in temperature will cause an increase in the diffusion of ions to the cathode. This can prevent the occurrence of uneven coating which appears as a vacuum of ions in the cathode. The appropriate temperature will tend to improve the quality of the coating.

Furthermore, using a magnetic field in a direction perpendicular to the electric field, namely in the direction of the anode-cathode, will increase the chemical reaction. This is because there is a flow of magneto hydro dynamics (MHD) induced in the solution. MHD is a conductive fluid that flows due to the effect of using an external magnetic field. The Lorentz force is one of the effects of MHD.

In Cu-Ni coating the presence of Lorentz force causes the direction of the Ni ion flow in electrolyte deviates from the original direction. Near the cathode, the presence of Lorentz effect leading infiltration of Ni ions in the oblique direction so that vacancies in substrate Cu which is not visible from the direction of the Cu surface becomes visible when viewed from the oblique direction. Therefore the Ni ions can occupy these vacancies. This makes the layer on the surface becomes more solid and microscopically it will be better than Cu surface at normal conditions. [11, 12].

2. Research Methods

2.1. Material research

The material used in this study consisted of copper plates with a size of 10×1.3 cm^2 of 5 pieces. This plate was used as a substrate and installed as a cathode. Nickel with the same size was used as an anode. Electrolyte solutions were made from a mixture of boric acid (H_3BO_3), nickel sulfate ($NiSO_4$), and nickel chloride ($NiCl_2$). Boric acid was used as a pH controller for electrolyte solutions consisting of nickel sulfate and nickel chloride. The distilled water was used to dilute the solution. The equipment consisted a plating bath which contains 300 mL electrolyte, a TCA-BTA thermocouple was used to control the temperature of the solution, electric stove to heat the electrolyte solution, the DC voltage source that was set at 1.5. volt, a PA214 Ohaus balancing to measure the mass of the plate before and after deposition, ultrasonic cleaner to clean the sample, and Multimeter to display the voltage and current on the four points probe.

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2.2. Method of experiment

In the electroplating process electrolyte solutions were prepared with the composition of H_3BO_3 (30 g), $NiSO_4$ (195 gr), NiCl2 (450 gr) and H_2O (750 ml). The solution is heated with Bunsen burners up to 40 °C, then the equipment is assembled according to Figure 1. The electroplating process is carried out at a voltage of 1.5 volts for 5 seconds with an electrode distance of 4 cm, and a magnetic field of

200 gausses. The electroplating process is repeated by varying the electrolyte temperature of 50 °C, 60 °C, 70 °C, and 80 °C.

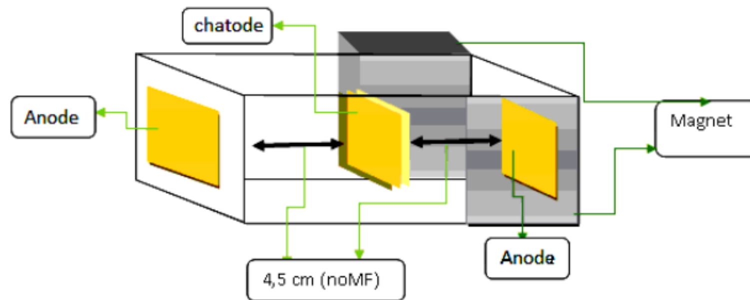

Figure 1. Scheme of equipment in the electroplating process

The mass of the substrate before and after electroplating was weighed to determine the thickness of the Ni layer following the equation:

$$d = \frac{\Delta m}{\rho A} = \frac{m_{\mathrm{Cu/Ni}} - m_{\mathrm{Cu}}}{\rho_{\mathrm{Ni}} A} \quad (1)$$

where d is the thickness of the Ni layer (cm), Δm is mass difference before and after coated (gr), m_{Cu} is mass of substrate Cu, $m_{Cu/Ni}$ is mass of Cu/Ni sample mass, ρ is Ni density (gr/cm3), and area A is Cu/Ni surface (cm^2). The errors of d are obtained from the propagation of errors from $m_{Cu/Ni}$, m_{Cu}, and A so that they become:

$$s_d = \sqrt{\left(\frac{1}{\rho A} s_{m_{\mathrm{Cu/Ni}}}\right)^2 + \left(\frac{1}{\rho A} s_{m_{\mathrm{Ni}}}\right)^2 + \left(\frac{m_{\mathrm{Cu/Ni}} - m_{\mathrm{Cu}}}{\rho A^2} s_A\right)^2} \quad (2)$$

Sheet resistivity was obtained by means of a four-point probes with the equation:

$$R_s = \frac{\pi}{\ln 2} \frac{V}{I} \quad (3)$$

If the measurement is done by variating I and recording the value of V, then Eq. (2) can be written as:

$$V = \frac{\ln 2}{\pi} R_s I \quad (4)$$

If it is assumed that $y = V$ and $x = I$, then by doing a linear regression on the data set (Ii, Vi) by following the equation $y = ax + b$ obtained:

$$a = \frac{\ln 2}{\pi} R_s \quad (5)$$

And then sheet resistivity is given,

$$R_s = \frac{\pi}{\ln 2} a \quad (6)$$

And the error of R_s is

$$s_{R_s} = \frac{\pi}{\ln 2} s_a \quad (7)$$

Where

$$s_a = s_{\hat{V}} \sqrt{\frac{N}{N\Sigma I_i^2 - (\Sigma I_i)^2}} \quad (8)$$

and

$$s_{\hat{V}} = \sqrt{\frac{\Sigma\left(V_i - \hat{V}_i\right)^2}{N-2}} \quad (9)$$

Where N the amount of data [12].

3. Results and discussion

The weighing Cu substrate and Cu/Ni sample are listed in table 1. Then in column 4 the data of weight of Ni was obtained using equation (1) with data ρ_{Ni} =8.91 g/cm^3 and the cross-sectional area of sample A = 7.61 cm^2.

Table 1. Nickel mass and the thickness at various electrolyte temperatures

Electrolyte Temp. (°C)	Mass of Cu (g)	Mass of Cu/Ni (g)	Mass of Ni (g)	Thickness of Ni (μm)
40	4.3531	4.3591	0.0060	0.09 ± 0.43
50	4.1126	4.1292	0.0166	0.24 ± 0.43
60	4.111	4.1323	0.0213	0.31 ± 0.43
70	3.5062	3.5322	0.0260	0.38 ± 0.43
80	4.0827	4.1144	0.0317	0.47 ± 0.43

In Figure 1 a curve of the relationship between the thickness of the Ni layer and the temperature of the solution is displayed.

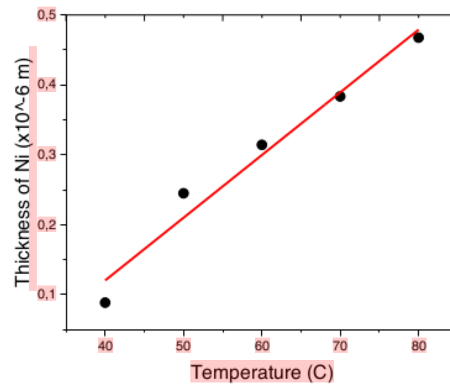

Figure 2. The thickness of the Ni layer at various electrolyte temperature

The thickness and the electrolyte temperature have a linear relationship

$$d = 0.009\ T - 0.243\ (\mu m) \quad (10)$$

with index terminated $R^2 = 0.97$.

Figure 1 shows that the thickness of the Ni layer increases with a higher electrolyte temperature. This is because the temperature has an influence on the rate of reaction. The higher the temperature, the faster the reaction rate. The increase in the reaction rate causes an increase in the kinetic energy of the ion so that the Ni ions move towards the cathode faster. With the high kinetic energy, the layers formed tend to be thicker [13,14]. The magnetic field here has a role to produce a layer with morphology that is formed to become finer.

Furthermore, the data of sheet resistivity of Cu before and after plated with Ni, and the resistivity difference between Cu and Cu/Ni is showed in table 2.

Table 2. The increasing sheet resistivity of Cu after plated with Ni

Electrolyte Temperature (°C)	R_s Cu (x10^{-3} Ω/sq)	Rs Cu/Ni (x10^{-3} Ω/sq)	ΔRs (x10^{-3} Ω/sq)
40	1.21 ± 0.03	1.34 ± 0.02	1.33 ± 0.02
50		1.33 ± 0.03	1.23 ± 0.03
60		1.32 ± 0.03	1.10 ± 0.03
70		1.30 ± 0.02	0.98 ± 0.02
80		1.29 ± 0.04	0.83 ± 0.04

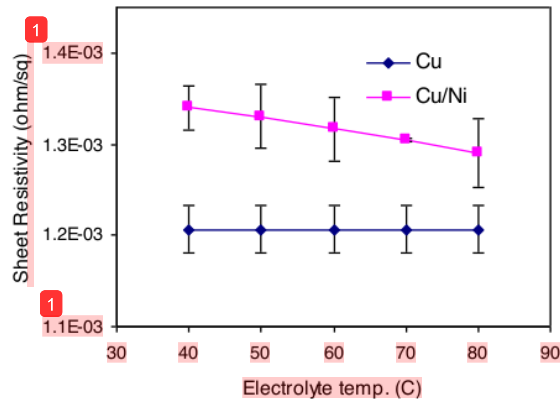

Figure 3. The sheet resistivity of Cu and Cu/Ni

Furthermore, Figure 3 shows the resistivity of Cu/Ni and Cu. The sheet resistivity of Cu/Ni decreases with the higher electrolyte temperature. The decreasing resistivity is caused by the increasing thickness of the Cu/Ni layer. Because nickel is a metal, the thicker the Ni layer that attaches to the Cu substrate, the Ni layer becomes more continuous, so that the sheet resistivity becomes smaller. Here, the electrolyte temperature significantly affects the diffusion of Ni ions that leading to the Cu cathode. If the temperature of the electrolyte solution gets higher, the solution becomes runnier.

From the curve in Fig. 3 it appears that sheet resistivity (R_s) from Cu/Ni has a linear relationship to the temperature of the solution T according to

$$R_s = -1.259 \times 10^{-6}\ T + 0.001\ (\Omega/sq) \quad (11)$$

where the determination index $R^2 = 0.99$. With this equation, it can be estimated the value of R_s which is related to the temperature of the electrolyte solution T.

4. 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

This is the preliminary research from the main research concerning with Cu/Ni temperature sensor. The researcher expresses his deepest gratitude to the Ministry of Research, Technology and Higher Education of the Republic of Indonesia which has provided research funding through the 2019 Higher Education Advanced Applied Research (PTUPT) scheme.

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