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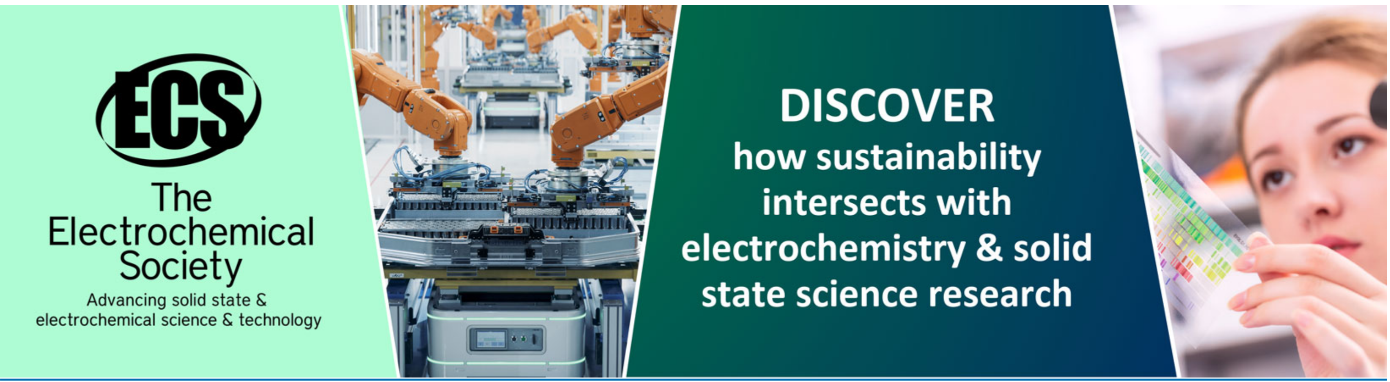

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Microstructure, thickness and sheet resistivity of Cu/Ni thin film produced by electroplating technique on the variation of electrolyte temperature

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Abstract. In this research, it has been made Cu/Ni thin film produced with electroplating technique. The deposition process was done in the plating bath using Cu and Ni as cathode and anode respectively. The electrolyte solution was made from the mixture of $HBrO_3$ (7.5g), $NiSO_4$ (100g), $NiCl_2$ (15g), and aquadest (250 ml). Electrolyte temperature was varied from 40°C up to 80°C, to make the Ni ions in the solution easy to move to Cu cathode. The deposition was done during 2 minutes on the potential of 1.5 volt. Many characterizations were done including the thickness of Ni film, microstructure, and sheet resistivity. The results showed that at all samples Ni had attacked on the Cu substrate to form Cu/Ni. The raising of electrolyte temperature affected the increasing of Ni thickness that is the Ni thickness increase with the increasing electrolyte temperature. From the EDS spectrum, it can be informed that samples already contain Ni and Cu elements and NiO and CuO compounds. Addition element and compound are found for sample Cu/Ni resulted from 70° electrolyte temperature of Ni deposition, that are Pt and PtO_2. From XRD pattern, there are several phases which have crystal structure i.e. Cu, Ni, and NiO, while CuO and PtO_2 have amorphous structure. The sheet resistivity linearly decreases with the increasing electrolyte temperature.

1. Introduction

In the husbandry industry, the existence of low-temperature thermometers is needed especially on the breeding using artificial insemination (IB). For the purposes of IB to obtain the qualified cow sperm the government is still importing. During delivery and storage, the sperm is placed in a special flask containing liquid nitrogen with the temperature of -198°C. As a part of the design of flask, there is a hole for liquid nitrogen to evaporate out to avoid the explosion. The problem is if the evaporation occurs continuously then the nitrogen content in the flask becomes less and the temperature increase. If the temperature inside the flask reaches the critical temperature i.e. -100°C then the cow sperm will die. Therefore, a low-temperature thermometer attached to the flask is needed.

Making low-temperature thermometers is not easy because it may make the equipment does not work as which should be such as freezing in the sensor so the sensor can not flow the current normally, freezing on conductor so that it can not function as a good conductor, the presence of mechanical stress, and others. These make a measurement on low-temperature need a long time compared to the measurement of medium in the room temperature [1,2]. The RTD (Resistance Temperature Detector) sensor is a sensor used to measure temperature by correlating RTD element resistance with temperature [3,4,5]. RTD elements that can be used are Platinum [6,7], Nickel [8],

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Copper [9,10,11], Balco, and Tungsten. But the elements are commonly used Platinum and Copper. The advantages of RTD sensor compared to others such as thermocouple, thermistor, bimetallic include a wide temperature range from -200°C to 800°C, more accurate than those, high stability, repeatability, and resistance to electronic noise, resistance to both corrosion and oxidation. Other advantages are a simple theory not as theory on IR thermometer involving the wave length and permittivity of measured medium, simple working principles, and cheap [12,13,14,15]. This will economically influence on cost savings.

The RTD elements are generally made from either metal or alloy, in the form of coils or thin films [16,17]. A good RTD sensor is capable to perform the large resistance changes to respond the temperature changes. A Cu film sensor can already perform as a temperature sensor, but the change of its resistance to respond the change of temperature is still limited. The resistance of Cu thin film can be increased when it is combined with nickel. Making of an alloy Cu-Ni thin film or Cu coated with Ni to form Cu/Ni is an alternative material for the low-temperature sensors. Both have several conformities, i.e. both are transition metal, both materials have the same type of crystal structure fcc, both materials have almost the same atomic size, i.e. 124 pm for Cu and 120 μm for Ni. These things facilitate the occurrence of combinations, or mutual substitution mechanisms between the two materials [18]. In addition, nickel is a good material when paired with copper because of the adhesion force on the interface between Cu and Ni strong [19].

Electroplating is one method to produce Cu/Ni film. In coating Cu plate with Ni, the temperature of the solution plays a role in facilitating Ni ions to penetrate the electrolyte solution to the Cu plate functioned as cathode. The higher electrolyte temperature the more Ni ions attach to Cu cathode. It is hoped that in the presence of Ni film on Cu plate, the resistivity of Cu/Ni film greater than Cu layer resistivity alone. But with the thicker Ni, Ni film becomes more continuous and thicker.

Therefore, the addition of Ni layer thickness will produce the Ni layer more conductive, otherwise becomes less resistive. So, after the first Ni coating, the resistivity of the Cu/Ni film is greater than that for Cu film alone, but with the rising electrolyte temperature, the resistivity of Cu/Ni layer will decrease. However, this film is expected to be more accurate in responding the changes of medium temperature.

2. The Experimental details

2.1. Materials and tools

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 is made from a mixture of borax acid (H_3BO_3), nickel sulphate ($NiSO_4$), nickel chloride ($NiCl_2$), and aquadest of 7.5g, 100g, 15g, and 250ml, respectively. Alcohol is used to rinse the substrate. Cleaning powder brand Rinso is used to clean the substrate of impurities and grease sticking, polisher brand Brasso is used to polish the substrate, liquid nitrogen is used as a low temperature medium that temperature will be measured by the sensor and its performance will be studied.

The equipment used for preparation the sample consisted of 1000 ml beaker glass wich is used as plating bath, benzene burner which is used to heat the electrolyte solution, vernier thermocouple is used to control the temperature of the solution stable at 60 °C, DC voltage set on 1.5 volts as power supply, PA214-type Ohaus balance is used to measure the plate mass before and after plating, ultrasonic cleaner is used to clean the sample, and two multimeters are used to measure the voltage and current on the four point probe tool. While the equipment for characterization is SEM-EDS to know the existence of Ni and its content and four-point probe to determine the sheet resistivity of the film. Temperature sensors (vernier thermocouple) and voltage sensors equipped with logger pro software is used to determine the output voltage of the sensor at various temperatures that can be seen on the computer screen.

2.2. Experimental procedure
In preparing the sample, the copper plate with the thickness of 0.2 mm cut with the size of 10 cm ×1.3 cm and then sanded gradually with abrasive paper number of 1200 CW, 1500 CW, and 2000 CW. Then the sanded plates polished with autosol, cleaned with rinso solution, rinsed with aquadest, and at the final stage rinsed with alcohol in the ultrasonic cleaner for 30 minutes. Samples that have been cleaned dried with a hair dryer, after drying wrapped in tissue, and stored in plastic clips.

In the electroplating process an electrolyte with compositions as pointed above prepared. Furthermore the equipment is set by placing Ni at the anode and Cu at the cathode. The circuit connected to a 1.5 volt voltage power supply, for 1 minute and a 4 cm electrode distance. The electroplating process carried out at temperature of 40°C, 50°C, 60°C, 70°C, and 80°C.

2.3. Methods of data analysis
In this case determination of Ni film thickness formed on Cu substrate was done by indirect measurement, that is measuring the mass difference before and after coated, area and density of sample according to equation [20,21,22],

$$D = \frac{\Delta m}{\rho P L} \tag{1}$$

where D = the thickness of Ni layer,

Δm = the difference of mass between Cu/Ni and Ni films.

P = sample length

L = sample width

ρ = density of Ni

The thickness of the obtained Ni layer is confirmed by the thickness of the Ni layer from one of the samples photographed in mapping element type. The determination of the existence of Ni and Cu film both element and compound are done by EDS (*Energy-Dispersive X-Ray Spectroscopy*). The measurement of sheet resistivity was done by four point probe equipment. The current flowing in the sample is varied so a set of voltage (V_i) and current data (I_i) is obtained. Furthermore, the data of V_i and I_i are fitted in a straight line so the equation becomes:

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

By assuming the graphic slope equal to a that is

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

then sheet resistivity is obtained as,

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

Analysis on the pattern of X-ray diffraction is done by observing the crystalline phase and the direction of the hkl crystal plane. Analysis of the effect of electrolyte temperature on the sheet resistivity is done by making graph of sheet resistivity to temperature. Furthermore, from the curve determined the relationship between the two variables.

3. Results and Discussion
In Figure 1 it is shown the image of Cu substrate to be coated by Ni. The substrate is divided into 2 parts, Figure 1(a) is Cu substrat that will be used to determine the sheet resistivity and SEM-EDS and Figure 1(b) is Cu substrat that has a litography will be used to study the behaviour of material as low-temperature sensor.

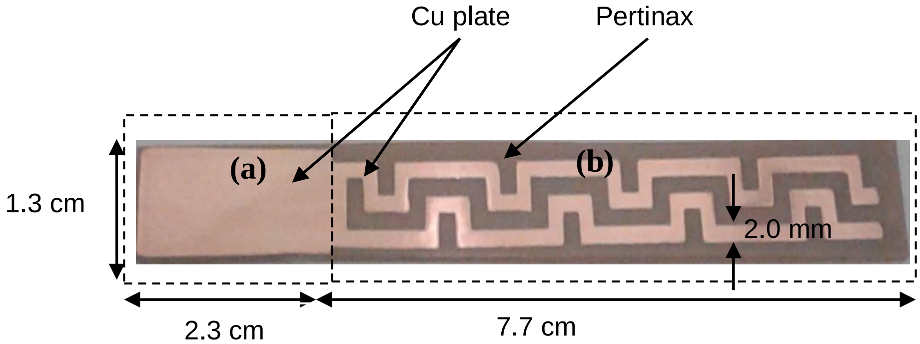

Figure 1. Lithography design of Cu substrate, (a) A part to be used as SEM-EDS analysis and determining sheet resistivity, (b) A part to be used as low-temperature sensors.

Table 1 shows the of current density in the plating process at various temperatures of the electrolyte. The current density as shown in column 2 Table 1 is obtained by measuring the electric current in the circuit using the amperemeter. The current flow occurs because the circuit is connected to a 1.5 volt voltage source. Since the current in the electrolyte solution flows between the Cu and the Ni electrodes having a cross-sectional area of 13 cm^2, the current density can be obtained from dividing the measured current by the area of the electrode cross section. The change of electrical current occurs because the temperature of the electrolyte changes.

Table 1. Current density for each rising of electrolyte temperature

Electrolyte temperature ^{o}C)	Current density (A/mm^2)
40	0.04
50	0.05
60	0.07
70	0.08
80	0.10

In Fig. 2 (a) it is shown a SEM photograph from surface of the Ni layer above the Cu substrate. This sample is resulted from Ni deposition at 60°C electrolyte temperatures, while Fig. 2 (b) is a SEM photograph of the surface of Ni layer resulting from Ni deposition at 70°C electrolyte temperatures. Photographs were taken for only 2 samples in order to know the qualitative differences in the surfaces of the two samples. From the two images, researchers have not been able to reveal the difference because there is no data obtained, except only presents the results of the photograph. Indeed, in Figure 2 (a) it has looked like islands and it are Ni materials. But in Figure 2 (b) the islands are not visible. Perhaps this is due to the poor sample surface preparation to be photographed.

In Fig. 2 (c) and Fig. 2 (d) a cross-sectional images of a Cu / Ni sample obtained from Ni deposition on a Cu substrate at electrolytic temperature of 60°C and 70°C are presented. Photos were taken with magnification of 1000x for the sample of Figure 2 (c) and 2000x for the sample of Figure 2 (d). The difference of magnification is due to technical reasons that are to obtain a clear picture, because the choice for the same magnification of one image is clearly visible, while it is for the other image looks blurred. Both photographs have not revealed any Ni layer attached to Cu substrate.

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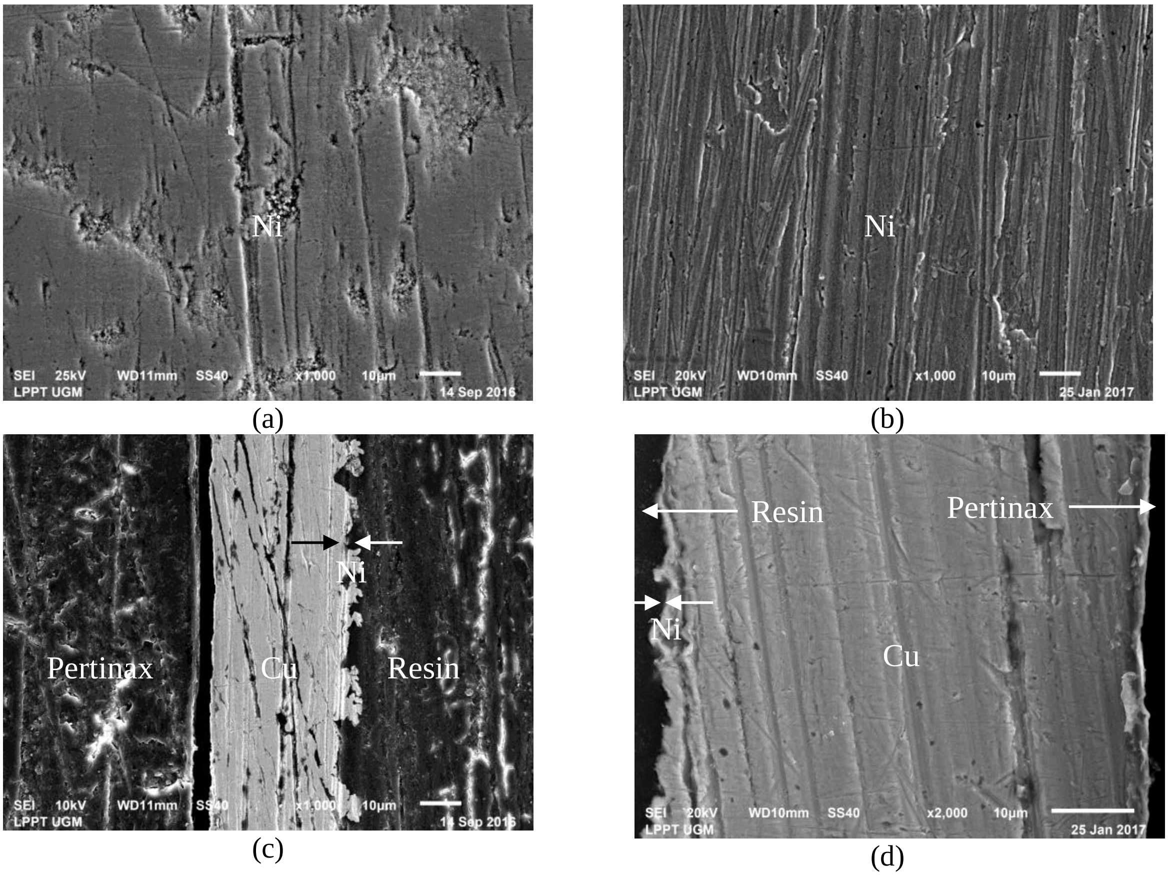

Figure 2. Image of the Ni film in the Cu/Ni sample produced from electroplating at: (a) 60°C and (b) 70 °C electrolyte temperatures.

Furthermore, the analysis was continued with EDS to determine the presence of Ni elements in the Cu/Ni sample and the content in the sample resulting from Ni deposition at 60°C and 70°C electrolyte temperatures. The results are shown in Figure 3.

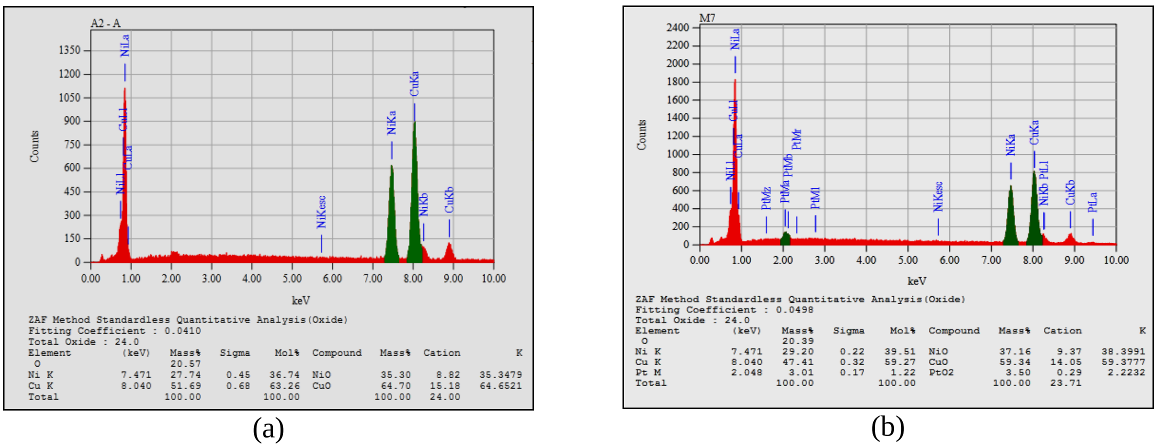

Figure 3. The EDS spectra of Cu/Ni samples resulting from Ni deposition on Cu substrate at electrolyte temperature of (a) 60°C dan (b) 70°C.

Table 2. Description of the mass composition of the elements and the mass composition of the compounds in the Cu/Ni samples

Element	Mass of Element (%)		Mass different (%)	Compound	Mass of Compound (%)		Mass different (%)
	Temp. 60 °C	Temp. 70 °C			Temp. 60 °C	Temp. 70 °C	
O	20.57	20.39	-0.18				
Ni	27.74	29.20	1.46	NiO	35.3	37.15	1.85
Cu	51.69	47.41	-4.28	CuO	64.7	59.34	-5.36
Pt	-	3.01	3.01	PtO_2	-	3.60	3.6

From the EDS spectrum it can be informed that samples already contain elements of Ni and Cu. In addition, in this sample also contains compounds NiO and CuO. Samples from Ni deposition at 70°C electrolyte temperature contain the addition element and compound that are Pt and PtO_2. From the description of the mass composition of the element and compound, it is known that the Cu/Ni sample resulting from the Ni deposition with the electrolyte temperature of 60°C has produced Ni element with the content (in percent mass) of 27.74%. This percentage rises to 29.20% or increases by 1.46% when the electrolyte temperature rises to 70°C. Although the increasing percentage is not too large that is 10°C, but if the temperature rises from 40°C to 80°C the increase of mass percentage of Ni is expected to be larger. This indicates that the electrolyte temperature has an effect on the speed of Ni layer formation on the Cu surface. The higher the electrolyte temperature, the easier the Ni ions penetrate the electrolyte so that the current density becomes greater. Due to the increasing of Ni content, the Cu content decrease from 51.69% to 47.41% or decreased by 4.28%.

The presence of NiO and CuO compounds is the result of a reaction between nickel and oxygen from the electrolyte. This oxygen comes from the solvent element of borax acid (H_3BO_3) and nickel sulphate ($NiSO_4$). Both of these materials are needed in the electroplating process. Borax acid is needed to control the electrolyte so that the electrolyte remains in base state, while nickel sulphate is a contributor of Ni ions to produce Ni layers on Cu substrate. In the electroplating process, this temperature variation also affects the content of NiO and CuO compounds. The content of NiO compound is increased from 35.3% (percent mass) to 37.15% or increased by 1.85% while the content of CuO compounds decreased from 64.7% to 59.34% or decreased by 5.36%. At Ni deposition at electrolyte temperature of 70°C is obtained another element that is Pt. The present of Pt is thought to be an element originated from Ni plates on the anode. To process Pt into Pt^{2+} it takes a power of 2.28 eV (corresponding to its ionization energy), while the magnitude of the voltage of the power supply in the electroplating process is 1.5 volts less than ionization energy. Therefore, the electrolyte temperature of 70°C contributes to the ionization energy of Pt through thermal energy, which then the Pt ion moves toward the Cu substrate together with the Ni ions to form the layer on the Cu surface. From the EDS spectrum it can also be known that at a temperature below 70°C Pt ions have not appeared.

Furthermore, in Table 3 it is shown the thickness of Ni layer for each electrolyte temperature. The thickness of Ni film is determined using equation (1) with a cross-sectional area of A = 7.61 cm^2 and density of Ni = 8.9 g/cm^3.

Table 3. Measurements of mass and determination of thickness of Ni film

No	Electrolyte temperature, T (°C)	Mass of Cu substrat, m_1(g)	Mass of Cu/Ni film, m_2(g)	The thickness of Ni film, D ($\times 10^{-6}$ mm)
1	40	3.1347 ± 0.00003	3.1369 ± 0.00003	32.5 ± 4.80
2	50	3.1023 ± 0.00003	3.1055 ± 0.00003	47.2 ± 6.96
3	60	3.7090 ± 0.00001	3.7125 ± 0.00003	51.7 ± 7.58
4	70	3.1454 ± 0.00001	3.1498 ± 0.00001	65.0 ± 9.40
5	80	3.2028 ± 0.00001	3.2076 ± 0.00001	70.9 ± 5.30

From Table 2 column 4, it can be seen that along with the increase of the electrolyte temperature, the Ni film formed thicker. To see the relationship between the thicknesses of Ni film to electrolytic temperature, in Figure 3 is showed data (D_{id}, T_i). The trend of the data follows a straight line, so that the data is fitted according to the straight-line equation $y = ax + b$ where $y = D$ and $x = T$.

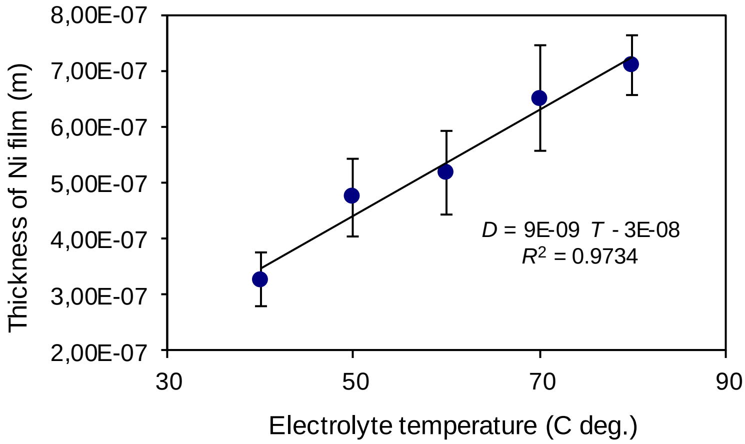

Figure 4. The curve of the relationship between the thickness of the Ni layer and the electrolyte temperature

The relationship between the electrolyte temperature (T) and the thickness of the Ni film (D) is,

$$D = 0.946\times10^{-6}T - 3.3\times10^{-5} \text{ (mm)} \quad (5)$$

This equation has a determination index R^2 = 0.97 so that the thickness of Ni film is strongly proportional to the electrolyte temperature. The higher the electrolyte temperature the thicker the Ni film.

To see the crystal structure formed on the sample, Figure 5 shows the X-ray diffraction pattern for the Cu/Ni sample resulting from deposition at various electrolyte temperatures. As a result, in Cu / Ni samples there are several phases which have crystal structure i.e. Cu, Ni, and NiO. The dominant peak of Cu occurs at a diffraction angle 2θ of about 43.3° with the orientation of the hkl plane (111) while the dominant peak of Ni occurs at a diffraction angle of about 39.04 ° with the orientation of the hkl plane (111). No Ni phase shift is caused by the variation of electrolyte temperature. The NiO phase appears at an angle of 41.5° with the orientation of the hkl plane (111), while the CuO and Pt, and PtO phases are not found. If this is confirmed by the element and compound resulted on the EDS spectrum analysis where on the EDS spectrum it is found Pt element, and CuO and PtO_2 compounds, we conclude that the Pt, CuO and PtO_2 are exist in not crystalline structure but amorphous.

The ordering of the crystal structure is related to the characteristics of the material, whether physical or electrical. In films that have a regular crystal arrangement will provide an easy flow of electric current to flow so that the resistivity is relatively smaller than the film that has the less regular or irregular crystal structure.

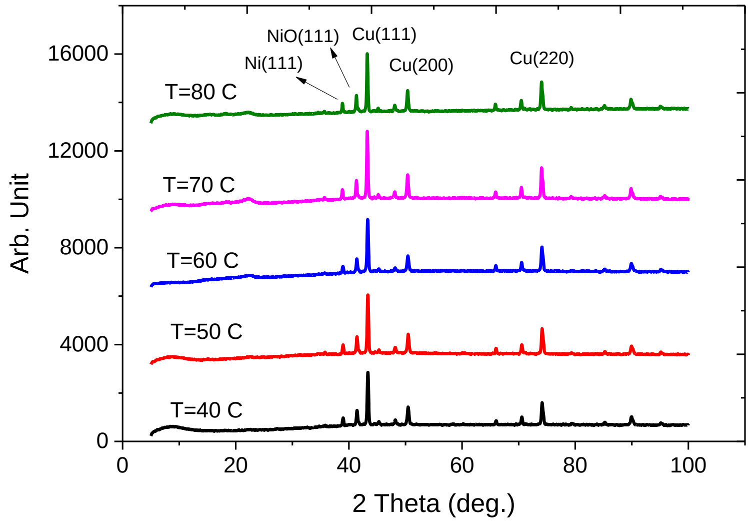

Figure 5. Pattern of X-ray diffraction for Cu/Ni samples resulted from Ni deposition at various electrolyte temperature.

Figure 7 shows the sheet resistivity of the Cu/Ni sample. Before coated with Ni, the resistivity of substrate Cu = (1,19 ± 0,08) × 10^{-3} Ω/sq and after coated with Ni the sheet resistivity of all Cu/Ni samples become increase greater than sheet resistivity of Cu substrate. This is in accordance with Afsarimanesh's expression that one of methods to increase Cu resistance is combining it with other materials to form alloys [9]. According to the Hume-Rothery formula if two materials have the atoms with nearly the same size, having a relative atomic size difference that less than 15%, having the same crystal structure, and the having an almost identical electronegativities, the two materials are easier to form the alloy because its are possible to diffuse substitutionally. In this process, the formation of this alloy is theoretically possible because the size of atoms of Ni and Cu is almost equal that are 0.1246Å for Ni and 0.1278Å for Cu, relative difference of the radius of the two atoms is 2.57%, while the crystal structure is the same i.e. fcc (*face centered cubic*), and its electronegativity is almost the same, that is 1.91 for Ni and 1.9 for Cu. So that there will be the diffusion process.

Table 4. The change of sheet resistivity before and after electroplating process.

No	Electrolyte temperature (°C)	$(Rs \pm S_{Rs}) \times 10^{-3}$ Ω/sq	
		Cu film	Cu/Ni film
1	40	1.150 ± 0.3	1.585 ± 0.09
2	50	1.234 ± 0.4	1.520 ± 0.08
3	60	1.197 ± 0.7	1.482 ± 0.08
4	70	1.204 ± 0.8	1.400 ± 0.08
5	80	1.145 ± 0.5	1.334 ± 0.07

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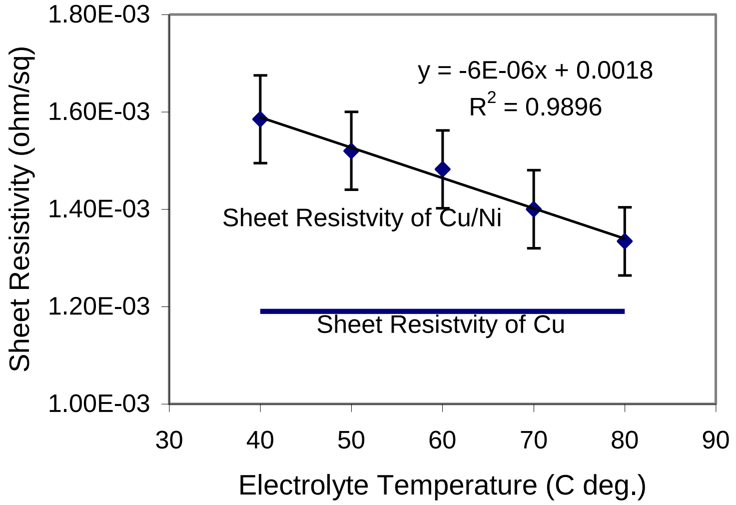

Figure 6. Sheet resistivity of Cu/Ni thin film on various electrolyte temperatures

In Figure 6 it appears that the sheet resistivity is inversely proportional to the electrolyte temperature. The relationship the two quantity follows a straight-line equation

$$R_s = -0.00006T + 0.0018 \quad (6)$$

Where T is electrolyte temperature and R_s is sheet resistivity. The value $R^2 = 0.9896$ indicates the temperature of the electrolyte has a strong effect on the sheet resistivity of the Cu/Ni film.

By knowing the profile of sheet resistivity on the electrolyte temperature variation, the Ni plating on Cu can increase the original Cu resistivity $(1.19 \pm 0.08) \times 10^{-3}$ Ω/sq to become larger, but with the increasing the Ni thickness due to increasing of the electrolyte temperature then the Cu/Ni resistivity becomes reduced. However, the Cu/Ni resistivity is still greater than Cu resistivity at all electrolyte temperature. According to the purpose of plating Ni to Cu is to increase the resistivity of the material so in this study the goal has been achieved. It is expected that with this achievement the research can be continued on the performance of Cu / Ni film as the low-temperature sensor.

4. Conclusion

In this research, we have succeeded in producing Cu/Ni films by electroplating method on the electrolyte temperature variation from 40°C up to 80°C. On the formation of Ni layer, the temperature variation affects the thickness of the formed Ni film. The thickness of Ni film corresponds linearly with the electrolyte temperature. The higher the electrolyte temperature the thicker the Ni layer. On the study on the the relationship between the electrolyte temperature to the sheet resistivity of Cu/Ni film, it is obtained the reverse linear relationship, i.e. the higher the temperature of the electrolyte the smaller the sheet resistivity. However, all of the resulted Cu/Ni films have a greater sheet resistivity than that for Cu film alone at all the variation of electrolyte tempera`tures. With this result, the study is expected to be continued on the sensor performance testing in measuring the low temperature medium.

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