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Effect of 30˚C Electrolyte Temperature on The Sensitivity Cu/Ni

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ABSTRACT

Given the necessity of cryogenic storage and monitoring cryogenic temperatures is equally important. This study aims to determine the resistance value and sensitivity of copper wire before and after electroplating at 30˚C electrolyte temperature as a low temperature sensor. The electrolyte solution consists of $NiSO_4$ 260 g, $NiCl_2$ 60 g, H_3BO_3 40 g and Aquades 1000 mL. Electroplating was carried out with an electrolyte temperature of 30˚C, electrode distance of 4 cm, voltage of 4.5 volts and plating time of 4 minutes. The plating results were analyzed to determine the resistance and sensitivity of the sensor at temperatures from 0 to -160˚C. The results showed that the resistance value of the Cu coil obtained R_{Cu} = (1.44 ± 0.00) ohm and the resistance of the Cu/Ni coil $R_{Cu/Ni}$ = (1.50 ± 0.00) ohm. The resistance value on the Cu/Ni coil (after plating) is greater than the Cu coil (before plating). While the test results of sensor sensitivity show that Cu and Cu/Ni coils have properties as low temperature sensors. Sensor sensitivity increases after plating. The sensitivity value obtained by Cu coil is $S(T) = -1E\text{-}06T + 6E\text{-}05$ and Cu/Ni coil $S(T) = -2E\text{-}06T + 2E\text{-}05$. The projection sensitivity at a temperature of -200 ˚C obtained is 0.00046 V/˚C less than the Cu/Ni coil 0.00082 V/˚C. So nickel plating on copper coil at 30˚C electrolyte temperature has successfully improved the sensitivity value of the low-temperature sensor.

Keywords: copper coil, Cu/Ni coating, electrolyte solution temperature, electroplating, sensitivity

INTRODUCTION

The ever-increasing development of technology in the 21st century has an impact on the food industry. In the past, the food preservation process used freezing techniques with ammonia. The coldness of the technology was low, making it easy to spoil food. From this limitation, the idea of cryogenics emerged. Cryogenics is a branch of physics that studies methods to achieve low temperatures far below room temperature [1]. This technology uses liquid nitrogen (LN_2) [2] as the refrigerant with a temperature of -196.1°C to -198°C [3]. Freezing with cryogenic technology is much better than conventional freezing techniques [4]. The freezing technology is used in preservation in various sectors including the livestock sector [5], the health sector and the food industry [6]. In food preservation, cryogenic methods can significantly reduce the formation of ice crystals compared to traditional cooling methods, so that the weight of the product does not increase significantly and the quality of the food is maintained. The cryogenic freezing method can also preserve animal sperm so that when it will be used for artificial insemination, the sperm is still normal with a motion index that is not reduced, so that fertilization of the egg will not fail.

With the development of cryogenics, a low-temperature measuring thermometer that works properly is needed. A temperature sensor is a component that can convert heat into electrical quantities, and temperature changes can be detected and analyzed using a specific circuit [7]. Special sensors that can be used to monitor temperatures down to -200°C are still bothering researchers in finding materials and manufacturing methods. Making such temperature sensors is not easy because the electrical and thermal properties of materials at this temperature are very non-linear [8].

One sensor technology that can work optimally at low temperatures is the Resistance Temperature Detector (RTD) sensor [8]. RTD is a sensor with a working principle using changes in thermal resistance influenced by temperature [9]. RTD elements are generally made of metal or alloy, in the form of rolls or thin films. Commonly used materials are platinum, nickel or copper. Platinum is the most commonly used constituent metal because it has a long term response and durability. However, platinum material is relatively expensive, so low-temperature measurement designs require higher costs. The abundant availability of copper wire encouraged researchers to try to study the response properties of copper wire to very low temperature changes. From a series of preliminary research experiences conducted previously, a feasible material for the cryogenic temperature sensor has been found, namely the combination of copper and nickel in the form of a thin layer of Cu/Ni or alloy [10].

Copper (Cu) has been used to measure very low temperatures because copper has good linear properties in responding to relatively small temperature changes [11]. Copper is able to respond to temperature changes down to -234.5°C. Copper wire formed into a coil is an effective form that can be used as a basis for low temperature measurement sensors [12]. Copper as an independent temperature sensor is still insensitive because of its small resistivity (16.78 nΩ.m at 20°C) but when paired with Nickel (Ni) which has a large resistivity (69.30 nΩ.m) it becomes a very sensitive sensor. In addition to increasing the resistivity of the coating using Ni, it can also increase the hardness of the material. Ni also has the ability to be used as a low temperature sensor with a range of -200°C to 320°C. Both materials may be able to form alloys because they have similar atomic sizes and crystal structures. Ni and Cu have almost the same atomic size of 0.1246 Å and 0.1278 Å and both have an fcc crystal structure, so that the diffusion process will form an alloy with a substitution bond type.

Besides alloying, the way to diffuse Cu and Ni metals is by a plating process. This method is known as electroplating. RTD sensors made with the electroplating method aim to form a dense thin layer on the surface of a metal using electrically assisted chemical electrolysis [13], [14]. The electroplating method was chosen because of its relatively cheaper price, fast, and easy control process [15-16]. Electroplating itself has received much attention from the industrial world and researchers because it offers many advantages such as fast production, very simple application process and low pollution [17].

Temperature in the electroplating process is a parameter that influences the microstructure of the composite layer because it is related to the increase in particle activity [18]. At too high a temperature the viscosity of the solution is reduced and there is a suppression of hydrogen gas activity. This will make it easier for Ni ions to penetrate the medium towards the cathode. As a result, the layer formed will be faster, thicker and uneven due to the risk of overplating. Therefore, with a temperature treatment that is not too high, it is expected to produce a thin and even layer so that a higher quality temperature sensor can be produced and can increase the sensitivity of the temperature sensor.

In terms of sensor sensitivity, the biggest part that contributes to sensitivity is the interface layer between Cu and Ni. If the layer is uneven, the layer is insensitive. The sensitivity of the sensor will increase if the layer is continuous and thin. If the thickness of the layer increases, the resistivity tends to decrease and the sensitivity decreases.

Cu/Ni low temperature sensors in the form of thin layers have been made at various variations, namely voltage [19], time [5,20-21], solution temperature [22-23], electrode distance [11], either using or without using a magnetic field. Referring to the results of the preliminary survey/experiment, it was found that copper wire formed into a coil shape is an effective form to create a large resistance value in the wire. The resistance of copper wire is also affected by the ambient temperature acting on it. The copper coil responds well to the low temperature changes produced by liquid nitrogen, so there is still an opportunity to increase the sensitivity of the sensor by nickel plating the copper coil. The advantage of the sensor in the form of a coil compared to that in the form of a thin layer is a very significant increase in resistance due to the dependence of resistance on the length of the wire and the small diameter of the wire. The temperature of the solution in the electroplating process is a very important factor [24]. It is important to start the electroplating process under simple conditions, including electrolyte temperature at room temperature around 30˚C. If this temperature is successful, it is hoped that it can be developed to other temperatures and the optimum temperature for the electroplating process can be obtained.

METHOD

The first stage in the thin layer manufacturing process is to prepare the necessary materials such as copper coils, nickel plates, and electrolyte solution consisting of $NiSO_4$ 260 g, $NiCl_2$ 60 g, H_3BO_3 40 g and Aquades 1000 mL. Electroplating was carried out with an electrolyte temperature of 30˚C, electrode distance of 4 cm, voltage of 4.5 volts and plating time of 4 minutes. The electrolyte solution was temperature controlled using an electric stove at 30˚C. FIGURE 1 shows the electroplating process of Cu and Ni.

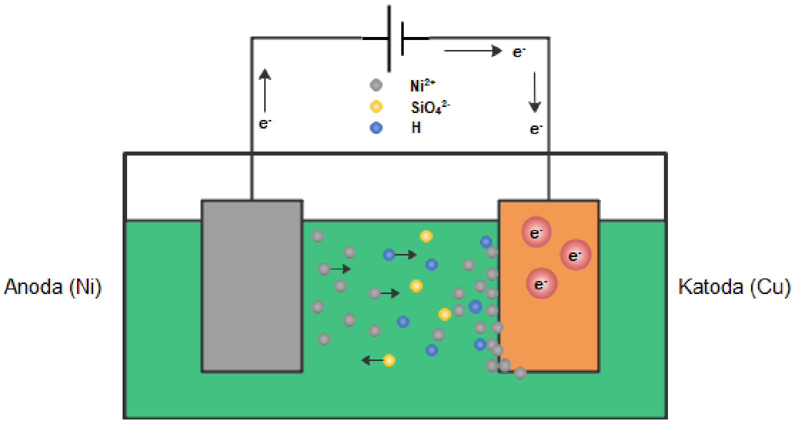


FIGURE 1. Electroplating Process

Analyzing the resistance value of the Cu and Cu/Ni coils is to calculate the resistance value (R) by summing the resistance value (R_i) divided by the amount of data (N) and calculating the standard deviation (σ). Calculations as in EQUATION 1 and EQUATION 2 below:

$$R = \frac{\sum R_i}{N} \tag{1}$$

and its standard deviation,

$$\sigma = \sqrt{\frac{\sum \left(R_i - \bar{R}\right)^2}{N}} \tag{2}$$

The results of the calculation of the resistance value of the Cu and Cu/Ni coils can be seen by using the percentage (%). The percentage (%) can be calculated from the initial resistance value (R_{initial}) which is the value of the coil before plating, namely Cu with the final resistance value (R_{final}) which is the value of the coil after plating, namely Cu/Ni. The calculation is as in EQUATION 3 below:

$$\text{Percentage}(\%) = \left|\frac{R_{\text{initial}} - R_{\text{final}}}{R_{\text{initial}}}\right| \times 100\% \tag{3}$$

The process of measuring sensor sensitivity is using testing tools such as laptops, transducers, Lab Quest Mini, voltage sensors, current sensors, LN_2 containers, and thermocouples. Sensor performance data is collected by slowly inserting and removing the Cu/Ni sensor along with the thermocouple in the LN_2 container. Sensor performance data will be displayed on the logger pro software. Determining the sensitivity of the sensor is by looking at the relationship between the RTD voltage value and the temperature change which is then processed with second-order polynominal data fitting [25], as follows:

$$y = ax^2 + bx + c \tag{4}$$

y is voltage (V), x is temperature (T),

$$V = (T) = aT^2 + bT + c \tag{5}$$

This method is used to test the sensitivity of the sensor where data is taken from the logger pro software which forms a non-linear graph. This can be generated by deriving the sensor sensitivity value from the equation of a second-order polynomial.

$$\frac{dy}{dx} = 2ax + b \tag{6}$$

So it becomes:

$$\frac{dV}{dT} = 2aT + b \tag{7}$$

The coefficients a, b, and c contain the x variable which is called the slope, meaning it shows the slope of the curve and can be used to determine the value of a function. While the coefficient d is called the intercept, which shows the point of intersection of the line with the y-axis. Manual calculations to determine a, b, c, and d. The smaller the value of a, the more linear the curve. Similarly, the greater the value of b, the more sensitive the sensor.

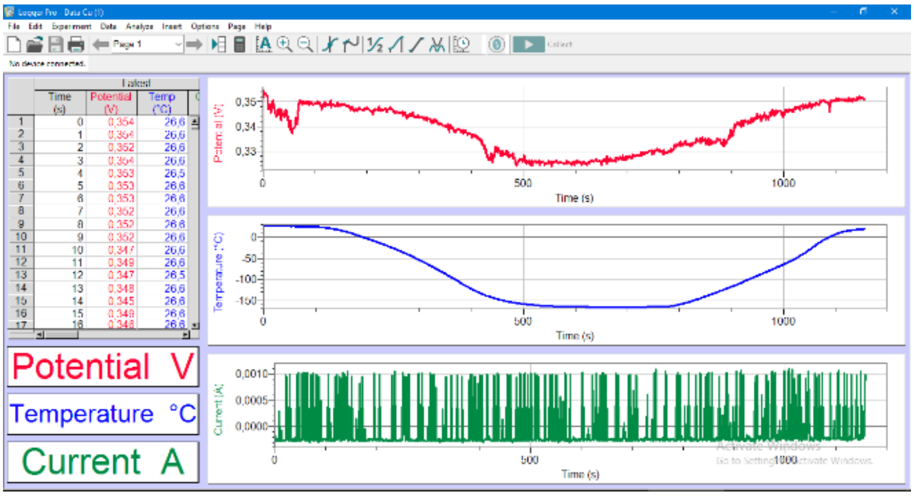
RESULT AND DISCUSSION

Cu/Ni coils were successfully deposited at a temperature of 30˚C, electrode distance of 4 cm, voltage of 4.5 volts and coating time of 4 minutes. The resistance values of Cu and Cu/Ni coils were then measured using a multimeter. There is a significant difference between the two data obtained from the experiment.

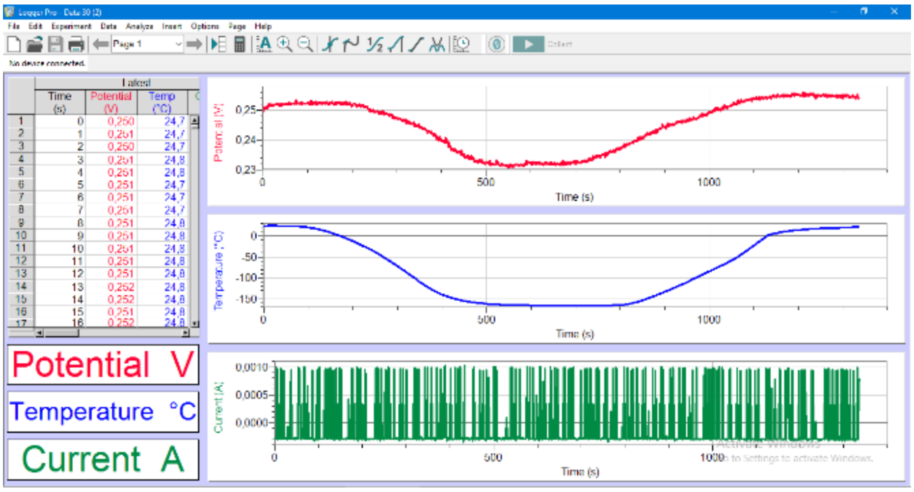
TABLE 1. Resistance Data

i	R_{Cu} (ohm)	$R_{Cu/Ni}$ (ohm)
1.	1.44	1.50
2.	1.44	1.49
3.	1.44	1.50
4.	1.44	1.50
5.	1.44	1.50
6.	1.44	1.50
7.	1.44	1.50
8.	1.45	1.49
9.	1.44	1.50
10.	1.45	1.50
Total	14.42	14.98
Average	1.44	1.50
Error	0.00	0.00

Based on TABLE 1, the Cu resistance R_{Cu}=(1.44±0.00) ohm and Cu/Ni resistance $R_{Cu/Ni}$=(1.50±0.00) ohm were obtained. From these results, using EQUATION 3, there is a difference of 3.8% and it can be seen that the Cu/Ni coil has increased.



(a)



(b)

FIGURE 1. Sensitivity test data (a) Cu coil and (b) Cu/Ni coil.

In FIGURE 2 the Cu coil shows that the graph already shows the characterisation of the sensor but the voltage produced is not stable. The lowest temperature reached by Cu and Cu/Ni is -165˚C. The response to temperature changes up to -165˚C Cu is faster. The time required for Cu to reach the lowest temperature is 476 seconds while Cu/Ni is 492 seconds.

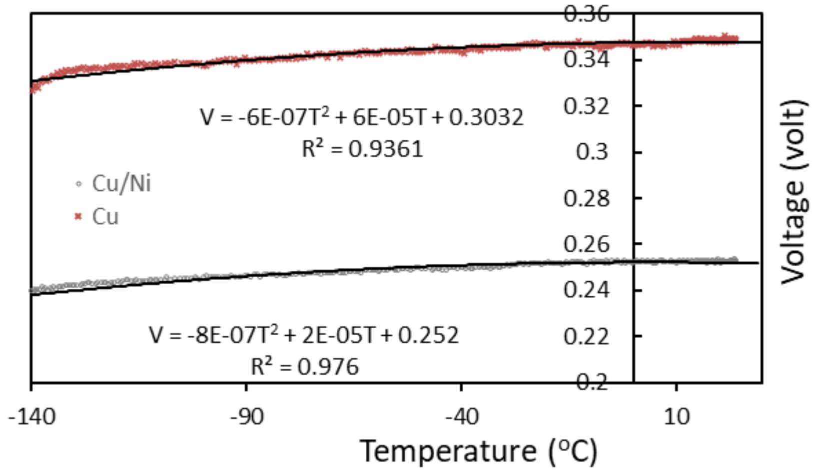


FIGURE 2. Graph of the relationship between voltage and temperature.

FIGURE 3 is a graph of the relationship between voltage and temperature of each coil. Data from the temperature is taken from normal temperature to temperature at the lowest point. Then the equation of second-order polynomial data fitting is obtained, as shown in EQUATION (8) and (9).

$$V_{(Cu)} = -6E - 07T^2 + 6E - 05T + 0.3032 \tag{8}$$

$$V_{(Cu/Ni)} = -8E - 07T^2 + 2E - 05T + 0.252 \tag{9}$$

In the Cu/Ni graph, a more regular curve is obtained than the Cu graph. The signal output from the Cu sensor tends to contain ripple while for Cu/Ni it is relatively smooth and stable. This is shown by the value of determination index R^2 is 0.9361 for Cu layer and 0.9760 for Cu/Ni layer. This proves that Cu coils have not shown good characteristics as low temperature sensors and Cu/Ni coils have shown characteristics as low temperature sensors. The lower the temperature, the smaller the voltage value. Because the greater the initial value of the voltage produced, the clearer the slope of the resulting curve.

The sensitivity of the sensor can be seen from the slope of the curve obtained from the graph of the relationship between sensor sensitivity and temperature. Because the curve is non-linear, a second-order polynomial data equation is used. Then the equation is calculated using the derivative of the voltage to temperature, namely S = $2ax + b$. The graph of the relationship between voltage and temperature in Cu and Cu/Ni clusters is shown in FIGURE 4.

The data obtained by the Cu/Ni coil is higher than the Cu coil data. It can be seen that the Cu coil sensor sensitivity is in the range of 0 to 0.00025 (V/˚C). While the Cu/Ni coil sensor sensitivity is in the range of 0.00005 to 0.00030 (V/˚C). The difference shows that the Cu/Ni coil, namely the coil after plating, is better used as a low temperature sensor and the resulting graph looks smoother and more stable between temperature changes and voltage changes.

From FIGURE 4, after plotting using the second-order polynomial equation, the sensor sensitivity results are obtained from the following second-order polynomial formula derivation as in TABLE 2.

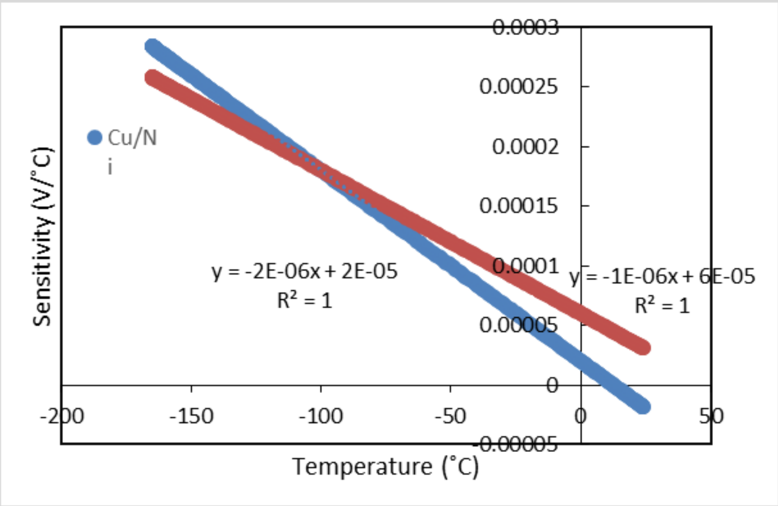


FIGURE 4. Graph of the relationship between sensor sensitivity and temperature for Cu and Cu/Ni.

TABLE 2. Sensor sensitivity test results.

Sensor Sensitivity (V/˚C)	
Cu	S(T) = -1E-06T + 6E-05
Cu/Ni	S(T) = -2E-06T + 2E-05

The derivation of the second-order polynomial equation which is the sensitivity value of the Cu and Cu/Ni coils is presented in TABLE 2. With *T* being the extrapolated temperature projected to -200˚C. This is done because the sensor can still reach lower temperatures, but the thermocouple can only reach temperatures of ±-165˚C. So that the sensitivity value can be obtained as in the following TABLE 3.

TABLE 3. T projection result on sensor sensitivity.

Sensor Sensitivity (V/C)		**Projection T=-200˚C**
Cu	S(T) = -1E-06T + 6E-05	0.00046
Cu/Ni	S(T) = -2E-06T + 2E-05	0.00082

The results of the T=-200˚C projection showed that the sensitivity value of the Cu/Ni coil has a greater value than the Cu coil. The Cu coil shows a value of 0.00046 V/˚C while the Cu/Ni coil is 0.00082 V/˚C. It can be concluded that the sensitivity value after plating is greater than the sensitivity value before plating.

CONCLUSION

From the research results of nickel plating on copper coils at 30˚C to increase sensitivity as a low temperature sensor, the resistance value of the Cu coil is obtained as R_{Cu}=(1.44±0.00) ohm and the Cu/Ni coil is obtained as $R_{Cu/Ni}$=(1.50±0.00) ohm. The resistance value of the Cu/Ni coil (after plating) is greater than that of the Cu coil (before plating). So the nickel plating on the copper coil has successfully increased the resistance value of the low temperature sensor. The sensor sensitivity test results show that Cu and Cu/Ni coils have properties as low temperature sensors. The sensitivity of the sensor increases after plating. The sensitivity value obtained by Cu coil is $S(T) = -1E\text{-}06T + 6E\text{-}05$ and Cu/Ni coil $S(T) = -2E\text{-}06T + 2E\text{-}05$. The result of the projection of T = 200 ˚C obtained that the sensitivity value of Cu/Ni coil has a greater value than Cu coil. The Cu coil shows a value of 0.00046 V/˚C while the Cu/Ni coil is 0.00082 V/˚C. So nickel plating on copper coil at 30˚C electrolyte temperature has successfully increased the sensitivity value of the low temperature sensor. So nickel plating on the copper coil at an electrolyte temperature of 30˚C has successfully increased the sensitivity value of the low temperature sensor. Since the electroplating process at a simple condition of 30°C electrolyte temperature has been successful, it is expected to be developed to other temperatures and can reach the optimal temperature for the electroplating process.

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