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Thickness Measurement and Sensitivity of Copper/Nickel Electroplating Results of Electrolyte Solution Temperature Variation

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ABSTRACT

Currently, cryogenic thermometers are needed and one of the uses of cryogenic thermometers is to measure the temperature of food preservation flasks. Research has been conducted on the manufacture of cryogenic thermometers derived from Cu/Ni coils by electroplating process with temperature variation treatment of electrolyte solution. The purpose of this study is to determine the effect of electrolyte solution temperature variation treatment on Ni thickness and Cu/Ni sensitivity as a low-temperature sensor. Electroplating was carried out with electrolyte temperature parameters of 30˚C-70˚C, electrode distance of 4 cm, voltage of 4.5 volts, and coating time of 4 minutes. The electrolyte solution was a mixture of $NiSO_4$ 260 g, $NiCl_2$ 60 g, H_3BO_3 40 g, and Aquades 1000 mL. Based on the results of the study, a remarkable condition was obtained on the thickness of Ni; namely, at 40 ˚C, the thickness increased to 1.08 μm. In addition, the best temperature can produce the greatest sensitivity value in Cu/Ni coil electroplating, namely at 50 ˚C.

Keywords: electrolyte temperature variation, electroplating, copper coil, temperature sensor

INTRODUCTION

The ever-increasing technological developments in the 21st century have significantly impacted the food industry. In the past, food preservation methods used freezing techniques with ammonia, which has a low chill rate, making it easy for food to spoil. However, from this limitation, an innovative idea known as cryogenics emerged. Cryogenics is a field in physics that focuses on methods to achieve extremely low temperatures well below room temperature [1]. This technology uses liquid nitrogen (LN2) [2] as its refrigerant with extremely low temperatures, ranging from -196.1 ˚C to -198 ˚C [3]. Compared to conventional freezing techniques, cryogenic technology in food freezing is much more effective [4]. The freezing technology has now been applied in various sectors, including as an organ preservation cabinet (the field of cryonics medicine), thermos gauges for storing semen (sperm) of animals that will be used for artificial insemination (animal husbandry) [5], and the food industry [6].

With the development of cryogenic technology advancing, there is an urgent need for a thermometer that can measure low temperatures with high accuracy. Researchers are facing a considerable challenge in finding materials and methods to manufacture specialized sensors capable of monitoring temperatures down to -200 °C. Making temperature sensors at this level is a complex task because the electrical and thermal properties of the materials at such temperatures are highly irregular [7].

One sensor technology that can operate optimally at low temperatures is the Resistance Temperature Detector (RTD) sensor [7]. RTD is a type of sensor that works based on changes in thermal resistance that are affected by changes in temperature [8]. RTD is a type of temperature measuring sensor included in the type of resistance thermometer [9][10]. RTD sensors are based on changes in resistance or resistance values due to changes in temperature [11][12]. When the temperature of the RTD element increases, the resistance of the element will also increase and vice versa. In other words, the temperature increase of the metal that becomes the RTD resistor element is directly proportional to its resistance. RTD elements are generally made of metals or alloys in coils or thin films. Commonly used materials are platinum, nickel, or copper. Platinum is the most commonly used constituent metal because of its responsiveness and long-term durability. However, platinum material is relatively expensive, so low-temperature measurement designs require higher costs. The abundant availability of copper wire encourages researchers to try to study the response properties of copper wire to very low-temperature changes. From a series of preliminary research experiences carried out by researchers and teams, a feasible material for cryogenic temperature sensors has been found, namely a thin combination of Cu/Ni or copper and nickel alloys [13].

Copper (Cu) was chosen as a material for measuring very low temperatures because it has a good linear response to relatively small temperature changes [14]. Copper can respond to temperature changes down to -234.5 ˚C. One effective form used as the basis of low-temperature measurement sensors is a copper wire formed into a coil or coil [15]. Copper as a temperature sensor independently is still less sensitive because the resistivity is small (16.78 nΩ.m at 20˚C). But, when paired with Ni, which has a large resistivity (69.30 nΩ.m), this can produce 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 can 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. Therefore, through the diffusion process, alloys can be formed with substitution bond types [16].

The RTD sensor is made by electroplating method which aims to form a dense thin layer on the surface of a metal using electrically assisted chemical electrolysis [17][18]. The electroplating method was chosen because of its relatively cheaper price and fast and easy control process [19][20].

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 [21]. The grain size of metal mass ions decreases slightly due to the increase in electrodeposition solution temperature [22]. Too-high temperature decreases the adsorption ability of the cathode so that the crystallization energy decreases, the density decreases, and the grains become coarse.

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, it is insensitive. The sensitivity of the sensor will increase if the layer is continuous and thin. If the layer thickness increases, the resistivity tends to decrease, and the sensitivity decreases. Therefore, there is an optimal time that results in the greatest sensitivity.

In 2013, researchers made low-temperature sensors made from copper coils with wire diameters varied from 0.1 mm to 0.2 mm. The number of turns is also varied from 3,600 turns to 12,000 turns to obtain optimum specifications [23]. The medium used is air cooled with liquid nitrogen to reach a temperature of 157 K then the temperature is increased to 253 K through evaporation. The results showed that at very low air temperatures, the susceptibility value is not affected by the number of turns and variations in the diameter of the winding wire because the medium in the solenoid is diamagnetic. The coil used in the study is copper material that has never been combined with coatings.

Similarly, Cu/Ni low-temperature sensors in the form of thin layers have been made at various variations, namely voltage [24], time [25][26][27], solution temperature [28][29], electrode distance [14], either using or without using a magnetic field.

There is still an opportunity to improve the sensor by nickel plating on a 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 wire length and small wire diameter. Therefore, in this study, further research is proposed by making Cu / Ni low-temperature sensors from nickel electroplating on copper coils. This research will be conducted with variations in solution temperature. The temperature of the solution in the electroplating process is a very important factor [30] because it affects the resistivity of the material [31] and corrosion behavior [32]. The temperature variation used is the solution temperature from 40 °C - 80 °C.

METHODS

Substrate Preparation

At this stage, the material prepared is copper wire with a diameter of 0.5 mm coming from a single cable. The copper wire is then cleaned by removing the wire from the outer layer of the cable. After cleaning, the copper wire is formed into a coil (winding) of 300 turns. Next, prepare the nickel plate and clean the surface using autosol until clean. Then, wash the plate using a toothbrush and rinse with soap. The nickel plate is rinsed again with distilled water and alcohol on an ultrasonic cleaner for 3 minutes. Then dried with a hair dryer and wrapped with tissue.

Preparation of Cu/Ni thin film

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. Materials to make the electrolyte solution were stirred using a magnetic stirrer for 3 hours. The deposition process was carried out by varying the temperature from 30˚C - 70˚C. Ni coating with an electrode spacing of 4 cm, voltage of 4.5 volts, and coating time of 4 minutes. Setting up a laptop to display the current on the pro logger. Setting the time on logger pro for 240 seconds for 1 data/second. Placed Cu on the cathode and Ni on the anode. Turned on the voltage source to dissolve the Ni particles. Turning on the voltage source along with collecting data on the pro logger. After the electroplating process is completed, wash the Cu/Ni coils with distilled water using an Ultrasonic Cleaner for 3 minutes. Then, wash the Cu/Ni coils with alcohol using an Ultrasonic Cleaner for 3 minutes. Drying the Cu/Ni coil with a hair dryer. Next, wrap the sterilized Cu/Ni coil with tissue and store it in a plastic clip.

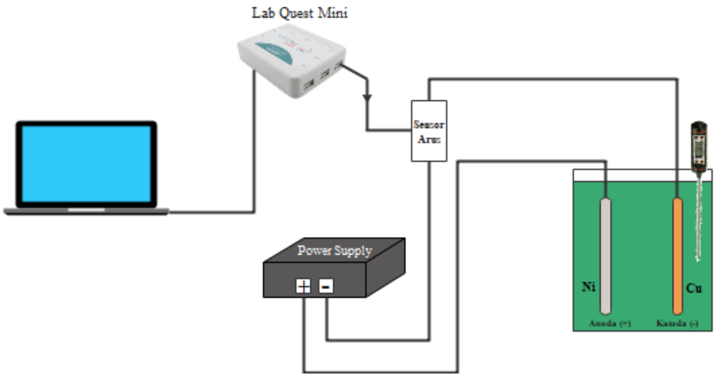

FIGURE 1. Tool set

Characterization of Cu/Ni coating

Determination of layer thickness

The process of measuring the thickness of the deposition layer formed in the electroplating process is calculated by weighing the sample before and after the electroplating process using an ohaus balance. Then, calculate the difference by reducing the weight before electroplating. The thickness of the Ni layer formed is calculated using the following equation.

$$D = \frac{W}{\rho A} = \frac{(m_{\mathrm{Cu/Ni}} - m_{\mathrm{Cu}})}{\rho A} \quad (1)$$

D is the thickness of the layer, W is the difference between Cu/Ni mass and Cu mass, ρ is the density of the Ni metal layer (gr/cm^3), which has a value of 8.908 gr/cm^3, and A is the sample area.

Sensor Performance Test

Cryogenic sensor performance testing was carried out in a medium with a temperature of -198 ˚C with the circuit, as shown in FIGURE 2. The test was carried out by immersing the sensor into LN2 and lifting it back up. The sensor calibration process is done with a thermocouple sensor. The data obtained were then analyzed to obtain sensor sensitivity, sensor response time, and sensor hysteresis.

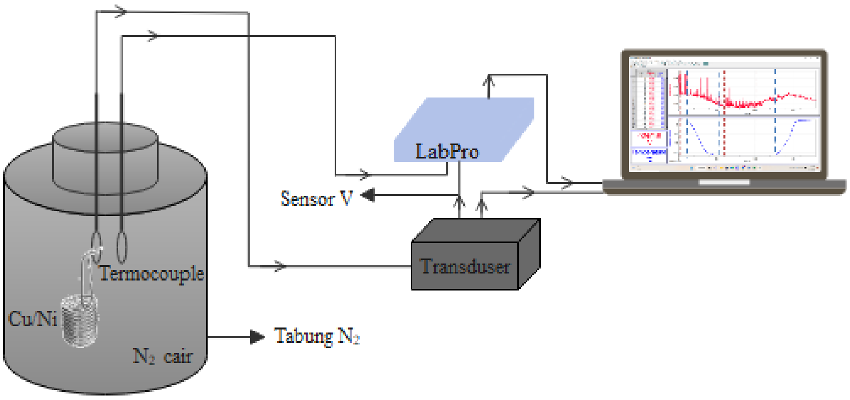

FIGURE 2. Sensor sensitivity test

Sensitivity is a measure of how much the sensor output changes with temperature. For outputs in the form of voltage, the sensitivity expresses the change in voltage to changes in temperature. The relationship between output voltage and temperature follows the Callendar-Van Dusen formula [33]:

$$R_T = R_0[1 + AT + BT^2 + (T - 100)CT^3] \tag{2}$$

A, B, and C are Callender-Van Dusen coefficients. While R_T is the resistance at 0˚C (Ω) and R_0 is the resistance at temperature T (˚C). The smaller the order of T, the better the sensor. Sensitivity is expressed by the derivative of R_T against T so that it becomes:

$$S(T) = \frac{dR_T}{dT} = R_0[A + 2BT + (4T - 300)CT^2] \tag{3}$$

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 polynomial data fitting [34], as follows:

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

where y is voltage (V) and x is the temperature (T),

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

This method is used to test the sensitivity of the sensor where the 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 that it becomes:

$$\frac{dV}{dT} = S(T) = 2aT + b \quad (7)$$

The coefficient values a, b, and c contain the variable x which is called the slope, meaning that it shows the level of slope of the curve and can be used to determine the value of a function. The coefficient d is called the intercept, which shows the point of intersection of the line with the y-axis. Manual calculation to find out 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.

RESULTS AND DISCUSSION

The results of Ni electroplating on Cu coils with variations in electrolyte solution temperature from 30 ˚C -70 ˚C, electrode distance of 4 cm, voltage of 4.5 volts, and coating time for 4 minutes resulted in the thickness of Ni deposits formed on Cu coils. The results of the sample mass before and after coating can be seen in TABLE 1.

TABLE 1. Sample mass data

Sample	Electrolyte Temperature (˚C)	m_{Cu} (g)	$m_{Cu/Ni}$ (g)	Δm (g)
1.	30	15.7410	15.7720	0.0310
2.	40	16.1410	16.2124	0.0714
3.	50	14.9085	14.9085	0.0000
4.	60	16.3014	16.3387	0.0373
5.	70	16.1259	16.1649	0.0390

The results of Ni thickness formed in each sample have different values. It was found that the thickness of the precipitate formed from 0.46 µm to 1.08 mm.

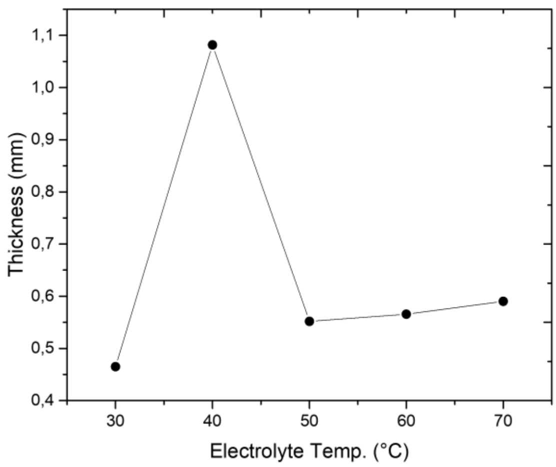

FIGURE 3. Thickness of Ni layer at various electrolyte solution temperatures

In FIGURE 3, there is a tendency that the higher the electrolyte temperature, the thicker the Ni layer. The temperature of the solution affects the rate of deposit formation because it makes it easier for the particles to reach the cathode. However, an extraordinary condition was obtained, namely at a temperature of 40 ˚C, the thickness increased to 1.08 µm. As many researchers have stated, 40 ˚C or a temperature between 40 ˚C and 50 ˚C is the best temperature for plating. By obtaining a Ni layer thickness of 1.08 µm, it is expected that this sample can behave as the best low-temperature sensor. High temperatures will produce coarse grains [22][35]. Too-high temperature decreases the adsorption ability of the cathode so that the crystallization energy decreases, the density decreases, and the grains become coarse. If the coating is uneven, the sensitivity will decrease. The sensitivity of the sensor will increase if the layer is homogeneous and thin. When the layer gets thicker, the resistivity tends to drop, and consequently, the sensitivity decreases.

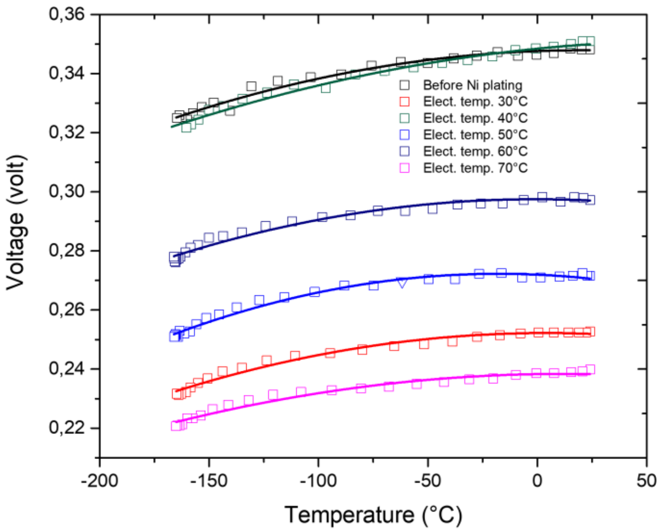

FIGURE 4. Relationship graph between voltage and temperature

The electroplating process with variations in electrolyte solution temperature applied to each sample is 30 ˚C to 70 ˚C with a range for each sample of 10 ˚C. FIGURE 4 is a graph of the relationship between the voltage and temperature of each coil. Data from the temperature is taken from normal temperature to temperature at the lowest point. While FIGURE 5 shows a graph of the relationship between sensitivity and temperature.

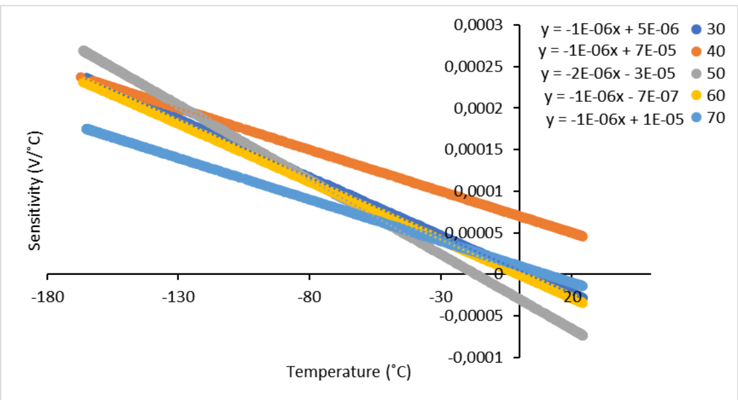

FIGURE 5. Relationship graph between sensitivity and temperature

Based on the analysis that has been done using EQUATION 7, the behavior results that there is the best temperature that can provide the largest resistivity value of the electroplating process shown in TABLE 1.

TABLE 2. Sensor sensitivity test results

Temperature (˚C)	**Sensitivity (V/˚C)**	**Projection T = -200 ˚C**
30	S(T) = -1E-06T + 5E-06	0.00041
40	S(T) = -1E-06T + 7E-05	0.00047
50	S(T) = -2E-06T - 3E-05	0.00077
60	S(T) = -1E-06T - 7E-07	0.00040
70	S(T) = -1E-06T + 1E-05	0.00041

Based on TABLE 2, there is a change in the sensitivity value of the Cu/Ni coil. The results of the T = -200 ˚C projection show that the sensitivity value of the Cu/Ni coil with 50 ˚C electrolyte temperature treatment has a greater value than the Cu/Ni coil at other electrolyte temperatures. The Cu/Ni coil at 30 ˚C - 50 ˚C increased until the electrolyte temperature of 60˚C decreased continuously. The largest sensitivity value at 50 ˚C shows a value of 0.00077 V/˚C. It can be concluded that there is the best temperature that can be used in electroplating Ni on Cu coils to produce the largest sensitivity value, namely at 50 ˚C.

CONCLUSION

Based on the results of Ni plating research on Cu coils with electrolyte temperature variations of 30 ˚C - 70 ˚C, a remarkable condition was obtained in Ni thickness, namely, at 40 ˚C the thickness increased to 1.08 μm. As many researchers have revealed, 40 ˚C or temperatures between 40 ˚C and 50 ˚C are the best temperatures for plating. In addition, the sensitivity value with an electrolyte temperature of 30 ˚C - 50 ˚C has increased. There is the best temperature that can be used in electroplating Ni on Cu coils that produces the largest sensitivity value, namely at 50 ˚C with a value of 0.00077 V/˚C. So temperatures between 40 ˚C and 50 ˚C are the best temperatures to carry out plating.

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