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EFFECTS OF ELECTROLYTE CONCENTRATION ON THE VOLTAGE RANGE AND RESPONSE TIME OF CU/NI FILM FOR LOW TEMPERATURE SENSOR

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ABSTRACT

The Cu/Ni thin film has been made with the electroplating method assisted by magnetic field parallel to electric field on the solution concentration variation. Electrolyte was made from the mixture of H₃BO₃, NiCl₂, NiSO₄, and H₂O with the content of each component is made more as many as 5 types those are C1-C5. Deposition is carried out using the Ni anode and the Cu cathode installed in the distance of 4 cm, the electrolyte temperature of 60 °C, 2 minutes under influence of 200G magnetic fields. The characterization was done on the voltage range and the sensor's response time. The result shows that the voltage range is inversely proportional to the response time. Sample of C3 has the smallest range of voltage and the longest response time, while sample of C5 has the greatest range of voltage and the shortest response time.

Key words: Electroplating, parallel magnetic field, Cu/Ni film, voltage range, response time

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1. INTRODUCTION

Copper (Cu) and Nickel (Ni) are good materials to be used as the low temperature sensor [1, 2] in the form of coil or thin film [3-5]. This sensor works is based on RTD (Resistance Temperature Detector) [6]. Electroplating is an easy and simple method to make Cu/Ni film. The electroplating is coating the metal surface by using chemical electrolysis aided by electricity [7, 8]. Among the advantages of the electroplating coating is corrosion resistance,

has a more aesthetic appearance, is more resistant to abrasion, is harder, and increased adhesion.

The use of the magnetic field (B) parallel to the electric field (E) in the electroplating process of Ni is to magnetize ferromagnetic of Ni in the solution, so Ni particles can be accelerated towards the cathode. This magnetic induction force is the kind of an external force that not influenced the electroplating parameters. The $B//E$ can make the distribution of Ni particles is more homogeneous [9] so the morphology of Ni deposit above the Cu surface is smoother [10, 11]. The use of $B//E$ can also increase the Ni ion transport to increase the current efficiency [12-14] so the viscosity of the electrolyte and the evolution of hydrogen gas can be reduced [15, 16]. It can accelerate the electroplating process accompanied with the good film microscopic structure [17]. In the electroplating, the electrolyte concentration determines the magnitude of the ion current [18, 19], and the electric current obtain the deposition rate. In Ni plating, the Watt nickel consists of boric acid, nickel sulfate, and nickel chloride dissolved. The use of boric acid is to keep the pH of the solution, while nickel chloride can prevent the pitting at the deposit surface. On the other hand, nickel sulfate together with nickel chloride can ease the anode solubility.

In this research, we study the voltage range and response time of Cu/Ni sensor produced by the electroplating on the variation of the electrolyte solution concentration. Plating process was assisted by the magnetic field parallel to the electric field.

Sensor that has the larger voltage range exhibits the more accurate. Furthermore, the response time towards the temperature changes is also a critical parameter in sensor. The faster the response the more real time the sensor. In fact, for the low temperature case, the thing is not ordinary. The lower the medium temperature, the slower the sensor in responding the temperature changes. This is related to the microstructure of the sensor materials such as the crystal regularity, the interplanar distance, the grain size, and external factors such as defects in the crystal and impurities.

2. MATERIALS & EXPERIMENTAL PROCEDURES

The Cu/Ni film was made by using the electroplating method. The materials used were commercial copper plate (Cu) and nickel (Ni) with dimension of (10.0 x 1.3) cm² placed as the cathode and the anode respectively. The electrolyte solution used in the deposition process consisted of H₃BO₃, NiSO₄, NiCl₂ and H₂O with the composition as written in table 1.

Table 1 Composition of electrolyte solution

Code	H ₃ BO ₃		NiSO ₄		NiCl ₂	
	(g)	(mol/L)	(g)	(mol/L)	(g)	(mol/L)
C1	30	0.484	225	1.461	40	0.313
C2	33	0.532	255	1.656	45	0.352
C3	36	0.581	285	1.851	50	0.391
C4	39	0.629	315	2.045	55	0.430
C5	42	0.677	345	2.240	60	0.469

Each component was put in the 1000 ml of distilled water then stirred with a magnetic stirrer for 6 minutes. Before being plated, the substrate was weighed by using an Ohaus balance of OHS-123 type and the weighed was noted as M_{Cu}. Next, Cu plate was installed as the cathode and nickel plate as the anode. The electroplating process was carried out in the voltage of 5.0 volt, the electrode distance of 4 cm, 2 minute time, the magnetic field intensity of 200 G, and the solution temperature of 60 °C. After finished, the sample was lifted up then

rinsed with distilled water and dried with a hair dryer. After dried, the sample was weighed again and the weight was noted as $M_{Cu/Ni}$.

The film characterization was done to find out the voltage range (in this context, the voltage is representation of temperature) and the response time towards the temperature changes for the decreasing and increasing temperature from 0 – -170°C and vice versa. The voltage sensor of VP-BTA together with thermocouple of TCA-BTA (acting as the calibrator) were immersed into the liquid nitrogen thermos with various temperatures from 0 – -170°C. The sensor immersion rate was 1.07 cm/minute. The outputs of this sensor were data of the voltage and temperature, and the graph of voltage-time and temperature-time.

To analyze the voltage range, the difference between the maximum voltages when the sensor is used to measure the temperature of 0°C and the minimum voltage when the sensor measures the temperature of -170°C was calculated. The response time of sensor was determined through fitting the voltage data towards time in accordance to the exponential equation [21]:

$$V(t) = k_1 + k_2 e^{-t/k_3} \quad (1)$$

where V is voltage, k_1 , k_2 and k_3 are constants. The value of k_3 is time constant which is the measure of time for the sensor to response towards the temperature changes. The bigger k_3 the faster the sensor in responding the temperature [20].

3. RESULTS AND DISCUSSION

Figure 1 shows the voltage-time curves of the Cu/Ni film as the deposition result on the variation of electrolyte concentration. All curves describe the sensor output voltages when used to measure the nitrogen temperature from 0 – -170°C and back to 0°C.

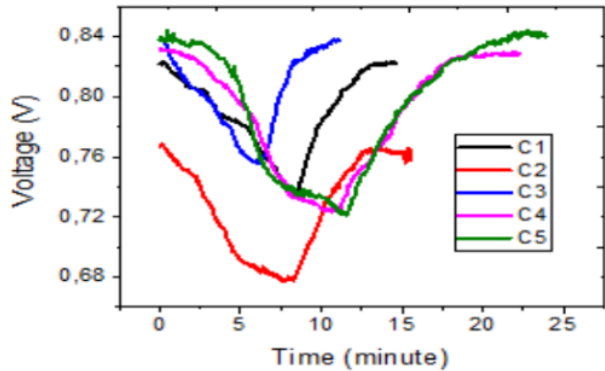


Figure 1 Voltage-time curves of Cu/Ni sensor as deposition result on solution concentration variation

The curves show that each sample has acted as a temperature sensor. The difference between one and another curve is on the starting voltage when the sensor starts measuring the temperature of 0°C. The concavity shapes of the ascending and descending curves, the value of minimum voltages of each curve, and the abscissa position from the minimum turning point of the curve.

3.1. Voltage Range

The voltage range is the difference between the maximum and the minimum values of the output voltage when the sensor is used to measure the liquid nitrogen temperature from 0 to -170°C. Figure 2 shows the voltage ranges of each sample.

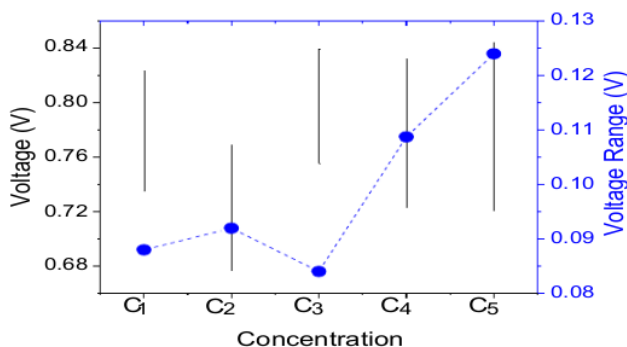


Figure 2 Voltage ranges of Cu/Ni sensor as deposition result on solution concentration variation

Figure 2 shows that the stem curves have different length. In addition, the starting points are also different. The difference in the starting points (position at the bottom of the stick) shows that the samples have different resistivity since the circuits are connected with the same power source so the current flowing in the circuits is also same.

Next, the dash curves show that generally the voltage ranges of the C1 to C5 samples are proportional to the electrolyte concentration. The greater the concentration, the greater the voltage range and vice versa. The difference of the voltage range is possibly due to the characteristics of deposit material produced from different electrolyte concentration. Except for the C3 sample the voltages do not proportional to the electrolyte concentration. The greatest voltage range is 0.084 volt. Confirmed with the analysis in the stem curves, the minimum voltage in stem of C3 is the greatest of all samples that is 0.76 V while other samples are lower than it. This shows that in this sample, the Ni film on the Cu surface is thinner compared to other samples so the sample has a high resistivity. If this sample is electrified when used to measure the medium temperature, the output is in the form of the voltage [22, 23]. On the contrary, if the Ni film gets thicker, so the Ni deposit will be more continues and it will decrease the value of its resistivity, so if it is electrified it will produce the low voltage. Unfortunately, the high resistivity of C3 causes the voltage ranges becomes small that is 0.084 volt. The C5 sample has the greatest range value of the output voltage that is 0.124 volt. The high-low output voltage range shows that the greater voltage range makes the Cu/Ni film sensor can response the greater temperature ranges so it has the more precise in appointing the temperature [24].

3.2. Sensor's Response Time

Response time is time needed by the Cu/Ni film sensor to response the temperature changes of the environment. For an environment of liquid nitrogen with the set temperature from -170 – 0°C, it can be identified the data of the sensor's output voltage change (representing the temperature) in each time. Figure 3 shows the result. It shows that the slope and the curvature of the curves are different where at times near the left ends (at the low temperature) the curves appear more sloping compared to the times near the right ends (near the temperature of 0°C)

the curves appear flatter. This means, on the temperature changes in the low temperature about -170°C , the sensor is relatively faster in responding the temperature changes compared to that in the environment with temperatures near 0°C .

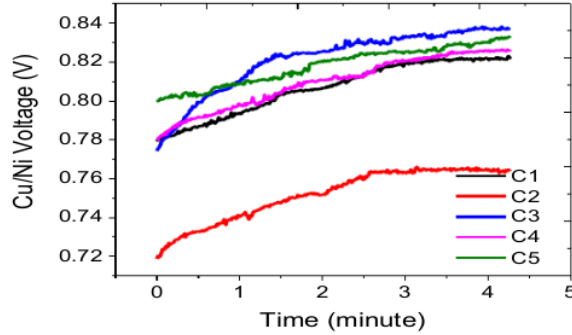


Figure 3 Response time of Cu/Ni film sensor

To obtain the fastest sensor in responding the temperature changes according with the equation (1), Table 2 shows the fitting constants from the data of voltage and time. Coefficient k_3 is the time constant. The bigger k_3 , the faster the voltage in reaching the maximum value.

Table 2 The fitting constants of exponential regression of voltage-time data for increasing temperature

Code	k_1 (volt)	k_2 (volt)	k_3 (s^{-1})	R^2
C1	0.8327 ± 0.0006	-0.0561 ± 0.0005	0.413 ± 0.010	0.99
C2	0.7659 ± 0.0003	-0.0472 ± 0.0005	0.719 ± 0.018	0.99
C3	0.8376 ± 0.0002	-0.0619 ± 0.0003	0.877 ± 0.011	0.99
C4	0.8311 ± 0.0002	-0.0519 ± 0.0002	0.488 ± 0.006	0.99
C5	0.8490 ± 0.0003	-0.0511 ± 0.0003	0.262 ± 0.004	0.99

Based on the result in Table 2 column 4, all graphs show the time needed for the samples to lift up from the bottom of the nitrogen thermos to the top with the speed of 1.7 cm/s (to obtain the temperature changes) is truly related to the sensor's output voltage exponentially. This is shown by the determination index of all samples that are more than 0.95 [25, 26].

Table 2 column 4 shows that time constants of all samples are approximately from $(0,262 \pm 0,004)/\text{s}$ to $(0,877 \pm 0,011)/\text{s}$. The value of k_3 is the biggest corresponding to C3 sample while the lowest corresponds to C5 sample. It is understandable that the C3 sample has the lowest time constant because the voltage range is the shortest, so it is quicker in reaching the peak of the curve. However, the time is the time needed by the sensor to reach the temperature of 0°C , so this sample is the fastest in responding the temperature changes but it does not provide the more accurate voltage reading scales. On the contrary, the time constant of the C5 sample is the lowest, but if it is confirmed with Figure 2, the voltage range is the greatest so this sample needs a little long time to reach the temperature of -170°C but more accurate since it provides the large voltage reading scales. The response time and the voltage ranges are inversely proportional. If the response time is long, the voltage range is short and vice versa.

4. CONCLUSIONS

This research has successfully made the Cu/Ni film with the electroplating method assisted by the magnetic fields parallel to the electric field on the electrolyte concentration variation. From the characterization of the voltage range and the response time to measure the liquid nitrogen temperature from -170°C to 0°C it can be concluded that the voltage range is inversely proportional to the sensor's response time. The C3 sample has the smallest voltage range but the highest response time. On the contrary, the C5 sample has the lowest/shortest response time but the greatest voltage range.

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