

Microstructure And Resistivity Of Cu/Ni/Cu/Ni (T=30 S 150 S) Thin Film Produced By Electroplating

By M. TOIFUR

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Abstract: The Multilayer Cu/ Ni/Cu/Ni has been made by electroplating method. In this study, the Ni₂ layer was made with variations in deposition time. The purpose of this study was to study microstructural studies with SEM-EDS test, X-Ray Diffraction, and multilayer resistivity values. The deposition process is carried out using two types of electrolyte solutions. In deposition Ni, the solution used is a mixture of H₃BO₃ 7.5 g, NiSO₄ 175, NiCl₂ 30 g. The Cu solution used was a mixture of CuSO₄ 62.5 g and 13 mL H₂SO₄. Each type of mixture is dissolved in 250 mL H₂O. The electroplating process is carried out at a voltage of 1.5 volts, the electrode distance is 4 cm. The solution temperature was 30° C for Cu deposition, 60° C for Ni deposition. Based on the results of the study it is known that the increased deposition time causes the particle size to be larger. Microstructure analysis shows that grain size, intensity, and interplanar distance influence the resistivity produced.

Keyword: Resistance Temperature Detector, Electroplating, Multilayer, Deposition Time, Microstructure

1 INTRODUCTION

Copper is a metal that can be used to detect low temperatures up to -234.5 [1,2,3]. However, pure copper metal is easily corrosive and has a smaller resistivity value of 1.7 μΩcm compared to nickel 7.3 μΩcm. Nickel can also be used as a temperature sensor [4]. By utilizing the physical properties of both copper and nickel materials, the two metals can be made into a sensor based on a Resistance Temperature Detector (RTD). RTD is a temperature detection device that utilizes the resistance value of a metal [2,5]. To increase the sensitivity of a sensor, copper, and nickel can be made into CuNi alloy, Cu/Ni, and Cu/Ni/ Cu/Ni thin layers. Multi-layers of Cu/Ni/Cu/Ni can be made by electroplating method. Electroplating is the best method for making thin layers, compared to other methods, because the method is easy to do and has a low cost [6]. Multi-layer synthesis aims to increase strength. Multi Layer will have various resistivity values. By making thin layers in multilayers, it is expected that the resulting sample has a good sensitivity value in response to temperature changes.

2 EXPERIMENTAL PROCEDURE

Detailed submission guidelines can be found on the author The material used in this study is a 10 x 1.3 cm Cu plate used as a substrate. Cu coating process uses a Cu plate 10 x 2 cm as an anode. Ni coating process uses 10x2 cm Ni as anode. The Deposition process is carried out in the following 2 stages:

2.1 Ni Deposition

The electrolyte solution used is a mixture of H₃BO₃, NiSO₄, NiCl₂, 7.5 g, 175 g, 30 g respectively. The material is dissolved in 250 ml aquadest. Electrolyte solutions are set at 60 °C.

2.2 Cu Deposition

The electrolyte solution used for Cu deposition was 62.5 g CuSO₄ and 13 ml H₂SO₄. The ingredients are dissolved in 250 ml aquadest. Electrolyte solutions are set at 30 °C. Cu and Ni deposition process were carried out at 1.5 DC voltage and 4 cm electrode distance. The coating process is carried out in three stages. The first stage is Ni coating on Cu or Cu₁/Ni₂. This process is carried out for 1 minute. The second stage is coating Cu against the Ni or Cu₁/Ni₁/Cu₂ layers. The Cu coating stage is carried out for 30 seconds. The third step is re-coating the Cu layer with Ni or Cu₁/Ni₁/Cu₂/Ni₂ layers. The third stage is carried out with variations in deposition time from 30 seconds to 150 seconds at 30 s intervals. Thin layers were characterized using several tests. Microstructure test with SEM photo aims to determine the morphology of the formed Cu/Ni/Cu/Ni layers. EDS analysis aims to see the percentage of Ni content in the sample for each variation in deposition time [7]. Microstructure tests using XRD were carried out to see the crystal structure, grain size, and d-spacing [8,9]. The resistivity test aims to determine the value of resistivity by using a four-point probe. The resistivity test is carried out at each coating stage.

2 EXPERIMENTAL PROCEDURE

SEM images are shown in Figure 1. The figure shows the surface morphology of each layer of Ni₂ deposit. In Figures 1a and 1b, it appears that the surface layer is not homogeneous. From SEM images, it can be seen that particles formed at 30 s deposition are smaller and more clustered than particles formed at 150 s deposition.

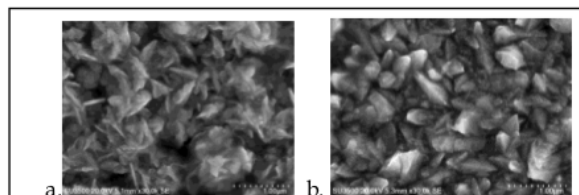


Fig. 1. . SEM images of surface morphology in Cu/Ni/Cu/Ni samples with a. 30 s Deposition time of Ni₂ and b. 150 s Deposition time of Ni₂.

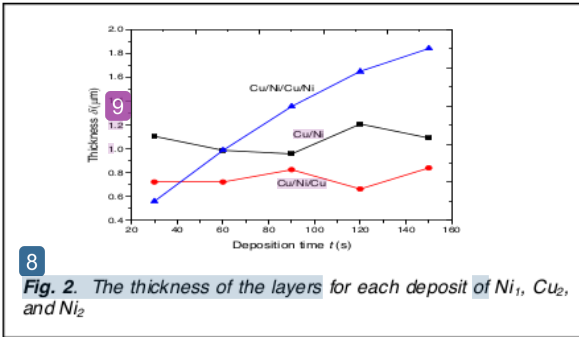
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From the EDS analysis, it is known that the variation in deposition time increases the Ni content as shown in Table 1. Ni content increased by 45.87%. These results indicate that deposition time affects the amount of Ni deposits.

5 **TABLE 1**
COMPARISON OF CU AND NI MASS IN CU/Ni/CU/Ni LAYERS

Deposition time (s)	Weight %	
	Cu	Ni
30	50.76	49.24
150	4.89	95.11

Furthermore, to determine the thickness of each deposited layer, mass measurements were carried out at each layer. The thickness values for each deposit of Ni and Cu are shown in Figure 2. In Figure 2 it can be seen that in the Ni₁ deposition stage, Cu/Ni samples have a thickness of ±1.10 μm, while Cu/Ni/Cu samples have a thickness of ±0.75 μm and samples of Cu/Ni/Cu/Ni samples have values that change according to the variation in time of deposition and tend to have linear changes.



8 **Fig. 2.** The thickness of the layers for each deposit of Ni₁, Cu₂, and Ni₂

The Cu₂ Ni deposit has a smaller layer thickness than Cu/Ni samples. This is due to different deposition times. If observed in Figure 2, Cu/Ni and Cu/Ni/Cu samples for each sample have different thickness changes. Changes in Cu/Ni layer thickness tend to be inversely proportional to changes in Cu/Ni/Cu layer thickness. Each Cu/Ni sample has a non-uniform thickness even though all deposition processes are carried out under the same conditions. This also happened in the Cu₂ deposition process for each Cu/Ni/Cu sample. This is probably due to the different substrate surface in each sample. Furthermore, in the microstructure test with X-Ray diffraction, diffraction peaks for each sample are shown in figure 3. In figure 3 it is known that the highest peak is at the angle ±43° in direction (111), while in direction (220) it is seen that Ni peaks formed in samples with deposition times of 90 s to 150 s. The dominant peak is in the direction (111).

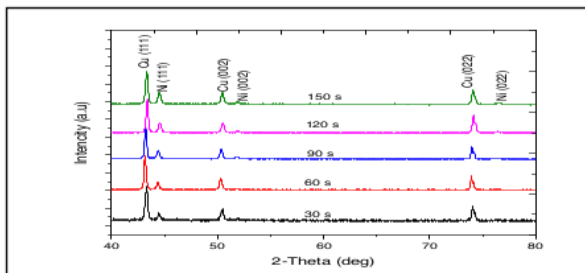


Fig. 3. Diffractogram for Cu/Ni/Cu/Ni samples

Furthermore, for interplanar distance in Cu and Ni metal is shown in Figure 4. In Figure 4, it can be seen that Ni has different interplanar distances and tends to decrease, except for with Ni₂ deposition samples for 60 s deposition.

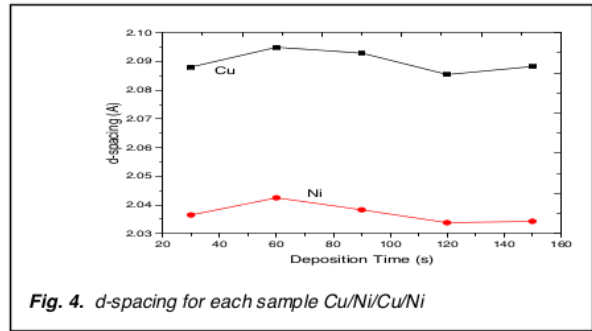


Fig. 4. d-spacing for each sample Cu/Ni/Cu/Ni

While in Figure 5, the intensity values for each sample at the diffraction peak are displayed at an angle of ±43°. In the figure, it can be seen that the Ni intensity value tends to increase except for 120 s deposition time. Figure 6 shows a graph of the relationship between the grain size and the time of deposition. Grain size tends to decrease. There is a relationship between the spacing in Figure 4, the diffraction intensity in Figure 5, and the grain size in Figure 6. D-spacing and grain size have an inverse relationship to the time of deposition. The longer the time of deposition, d-spacing and grain size tend to decrease. In Figure 5 it appears that the diffraction intensity is inversely proportional to the time of deposition.

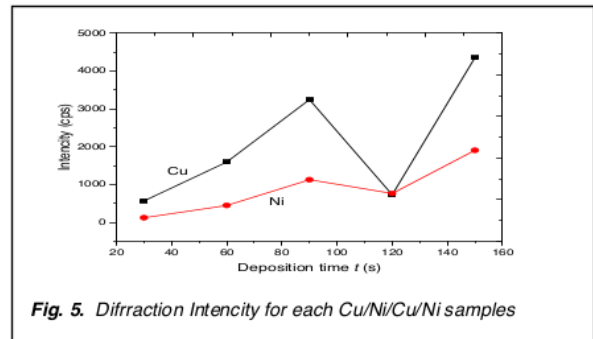


Fig. 5. Diffraction Intensity for each Cu/Ni/Cu/Ni samples

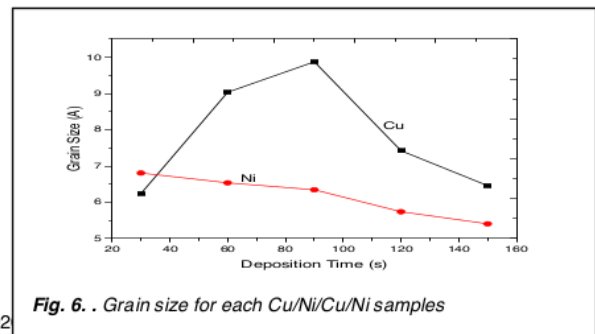


Fig. 6. Grain size for each Cu/Ni/Cu/Ni samples

Furthermore, the resistivity values in each sample are shown in Figure 7. From Figure 7 it can be seen that the samples of Cu, Cu/Ni, and Cu/Ni/Cu have different values for each sample. This is because the surface of each layer in the multi-layer Cu/Ni/Cu has a different morphology, causing different resistivity values.

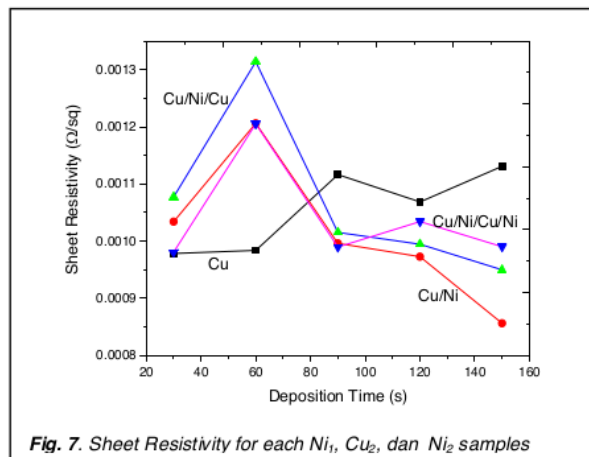


Fig. 7. Sheet Resistivity for each Ni₁, Cu₂, dan Ni₂ samples

In Ni₁ coating for Cu/Ni samples, the resistivity values increased in the 30s and 60s and subsequently decreased in the 90 s to 150 s. In Cu/NiCu samples the resistivity values increased compared to Cu/Ni for 30 s and 60 s and decreased from 60 s to 150 s. And for the last Cu/Ni/Cu/Ni samples, resistivity values at 30 s and 60 s have higher values than Cu substrate and have the lowest value compared to Cu/Ni and Cu/Ni/Cu samples. If observed in all deposition samples, the value tends to decrease except for deposition time 30 s.

4 CONCLUSION

Based on data analysis it is known that samples with multilayer coating can be done by electroplating. From microstructure analysis, the particle size of Cu/Ni/Cu/Ni samples is getting larger. From EDS analysis, the percentage of Ni is directly proportional to the time of deposition. While in the X-Ray Diffraction test it is known that the sample has a crystal structure and has a value of d-spacing which is proportional to the grain size and resistivity but inversely proportional to the intensity of diffraction.

5 ACKNOWLEDGMENT

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