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MICROSTRUCTURE AND RESISTIVITY OF THE ELECTROPLATED NI AIDED BY THE MAGNETIC FIELD PARALLEL TO THE ELECTRIC FIELD ON THE DEPOSITION VOLTAGE VARIATION

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ABSTRACT

This research has developed Cu/Ni film aided by the magnetic field (B) to investigate the effects of the deposition voltage on the microstructure and sheet resistivity of Ni film. Deposition was carried out with electroplating technique on the voltages that vary from 1.5 V to 7.5 V, while electrolyte temperature of 60°C, electrode distance of 4 cm, deposition time of 1 minute. The magnetic field of 200 gauss was installed on the parallel direction to the electric field (E).

The results show that the deposition voltages from 1.5 – 4.5 V produce Ni film with thickness directly proportional to the voltage, while on the voltages over 4.5 V the thickness of the film decreases until a half of maximum thickness. Electroplating has also produced the crystal structured Cu/Ni film. The dominant peaks are on the 2 theta angles of approximately 44.18° and 52.38° that correspond to Ni[110] and Ni[111]. The deposition voltage also plays a role in changing the regularity level of the crystal structure both for Ni[110] and Ni[111], also influence the inter-planar distance (d-spacing) for both directions [110] and [111]. From the voltage 1.5 – 4.5 V, the d-spacing profile of Ni [110] and Ni[111] are similar, but on the voltages over 4.5 V, the d-spacing of Ni[100] crystal decreases while Ni[111] increases. In accordance with the grain size, in the voltages from 1.5 – 4.5 V the grain size tends to decrease, but over 4.5 V it tends to increase. The deposition voltages from 1.0 – 7.5 V produce the Ni sheet resistivity (R_s) of approximately between 6.43 – 15.11 μΩ/sq. The highest value of 15.11 μΩ/sq corresponds to the voltage of 3.0 V. The high d-spacing and the small grain size contribute to the high R_s. Mean while, the lowest value of 6.43 μΩ/sq corresponds to the voltage of 6.0 V. The high level regularity of crystal structure, the great d-spacing, and the big grain size contribute to the low R_s.

Key words: Cu/Ni Film, Electroplating, Parallel Magnetic Field, Micro Structure, Sheet Resistivity

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1. INTRODUCTION

Cu/Ni thin film is a film potentially made as a low temperature sensor from 0 – -200°C. In principal, Cu has the potential to be a temperature sensor [1], but Cu is still less sensitive towards the temperature change because the resistivity of Cu tends to be still very low that is 1.72×10-8 Ωm [2] and the characteristics of Cu is easily oxidized. The sensitivity of Cu can be increased by synthesizing it with Ni that has higher resistivity that is 7.3×10^{-8} Ωm in the form of Cu/Ni film [3]. Nickel also has a good adhesion force, so it eases the deposition process [4,5].

In this research, the synthesis of Cu/Ni was carried out with electroplating technique aided by the magnetic field parallel to the electric field on the deposition voltage variation [6]. The electroplating process was chosen because it is more economic, fast and the process is easily controlled. The use of the magnetic field on the parallel direction to the electric field is intended to produce the magnetic induction force considering that Ni is a ferromagnetic material so it can increase the mass-transport deposits [7,8], increase the homogeneity of Ni ion distribution, accelerate the substrate coating process [9-14]. With the magnetic field, the film formed has a stronger texture. This will influence the characteristics of the material including the material resistivity [15,16]. The deposition voltage is a primary parameter in electroplating that plays a role in determining the current density, so it can increase the ion current [17], as a result, a film with better structure and morphology can be obtained [18,19].

2. MATERIALS & EXPERIMENTAL PROCEDURES

2.1. Substrate Preparation

This stage prepared the commercial copper plate and the nickel plate (10×1.3 cm^2), a lithograph design by using cutting sticker, $FeCl_3$, and acetone. The copper plate was cleaned by scrubbing the surface with autosol metal polish in the circular direction. The next stage was printing the lithograph design on the substrate by dissolving the substrate with $FeCl_3$ and cleaning it with acetone. After the design was printed, it was then cleaned by smoothing the surface with the toothpaste, rinsing with aquadest and alcohol on ultrasonic cleaner. Next, the substrate was dried with a hair dryer, kept by packing it on tissues, put it in a clip plastic and kept it in the drybox.

2.2. Electroplating of Cu/Ni

Before plating, the nickel solution (Watt's nickel) consisting of $NiSO_4$ 260 g, $NiCl_2$ 60 g, H_3BO_3 40g and H_2O 1000 ml was prepared. The substances were stirred by using a magnetic stirrer for 3 hours. The Cu plate as substrate was weighed by using Ohaus-PA214 balance and the weight was noted as M_{Cu}. The Cu plate was installed on the cathode and the Ni plate on the anode on the distance of 4 cm. Electroplating was carried out on the voltages by varying the deposition voltages from 1.5 V to 7.5 V, with electrolyte temperature of 60°C for one minute. The magnetic field of 200 gauss was installed on the parallel direction to the electric field. After finishing, 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 $M_{Cu/Ni}$.

2.3. Characteristics of Cu/Ni Film

The thickness of the Ni film can be determined by using the mass difference in the sample of Cu/Ni and Cu substrate. The thickness of the Ni film is calculated through the Lowenheim equation.

$$\delta = \frac{W}{\rho A_{Ni}} = \frac{(m_{Cu/Ni} - m_{Cu})}{\rho A_{Ni}} \quad (1)$$

With, δ is the thickness of the film, W is the mass difference between the mass of Cu/Ni ($m_{Cu/Ni}$) and the mass of Cu (m_{Cu}), ρ is the density of Ni, and A is the sample surface area.

The micro structure test was carried out with the X-Ray Diffraction (XRD). From the XRD diffractograph, it can determine the phase structure formed is crystal or a-morph, Miller index, the regularity level of the crystal structure, the peak angle and the full width at a half of maximum peak, the grain size and the inter-planar spacing.

The sheet resistivity test was carried out with the homemade four point probe. From the curve of the sheet resistivity towards the deposition voltage, information about resistivity related to the test result of the Ni film microstructure can be obtained.

3. RESULTS AND DISCUSSION

The monitoring towards the electric current for 60 second electroplating process is shown in Fig. 1. The monitoring towards the electric current is essential because it plays a role in the mass transport of Ni ion in the solution towards the cathode. The higher the deposition voltage the higher the electric current.

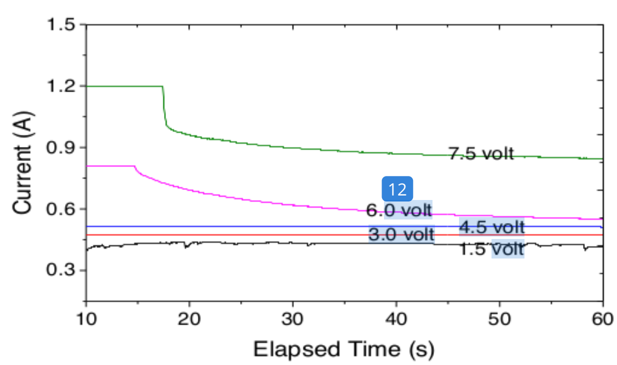

Figure 1 Electric current on electroplating process for various deposition voltages starting from 1.5 V to 7.5 V

For the voltage from 1.5 – 4.5 volt, the electric current tends to be stable, but on the voltages of 6.0 and 7.5 volt, the electric current tends to get decreased. This is due to the reduction reaction occurs near the cathode which able to creates hydrogen gas bubbles. As the bubbles are still near the cathode, they will inhibit the electric current.

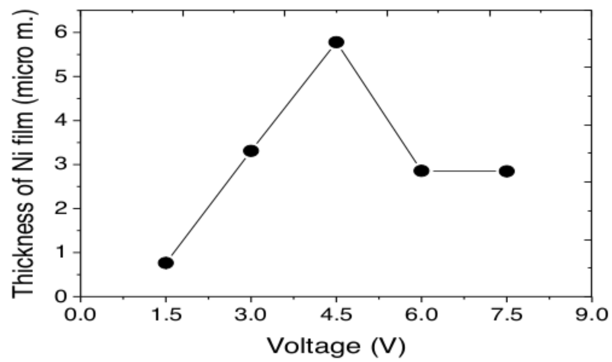

Figure 2 Effects of Deposition Voltage on Thickness of Ni film

The deposition voltage influences the thickness of the Ni film on the Cu surface. As shown in Fig. 2, the voltage variations produce the thickness between 0.76 – 5.78 μm. From the voltage 1.5 – 4.5 V, the thickness tends to increase. This shows that the deposition voltage, as disclosed by Fiqry et al, can increase the mass transport of Ni ion [20] so it obtains the thickness of Ni film that is proportional to the deposition voltage. However, over 4.5 V, the thickness gets decreased until 2.85 μm. It is suspected due to the occurrence of hydrogen evolution around the cathode during the deposition process. As disclosed by Chung et al [21], the main problem in electroplating is the presence of hydrogen gas around the cathode due to the water molecular reduction when the deposition process is running. This gas inhibits the electric current to reach cathode, so the plating process becomes useless. As a result, a film is not formed on the cathode surface.

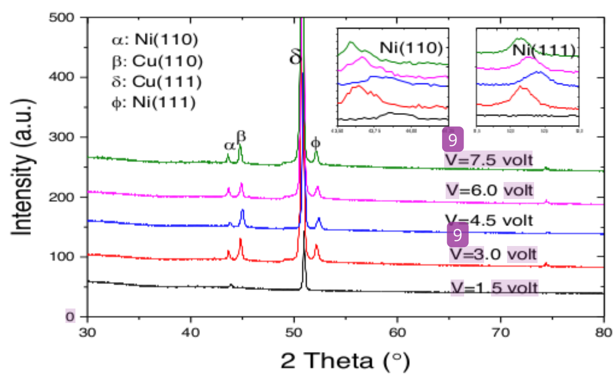

Figure 3 XRD Spectrum of Cu/Ni film on various deposition voltages

From the XRD spectrum, it obtains two dominant peaks for Ni those are Ni(110) and Ni(111) that occur on the angles of approximately 44.16° and 52.38°. With the increase of the deposition voltage, the peak angle position of Ni(110) tends to shift left, while for the peak angle of Ni(111) tends to shift right at first then shift left, as shown in sub-graph. The diffraction angle shifting to left or right will influence other microstructure parameters such as the inter-planar distance and the grain size, as discussed at the following part.

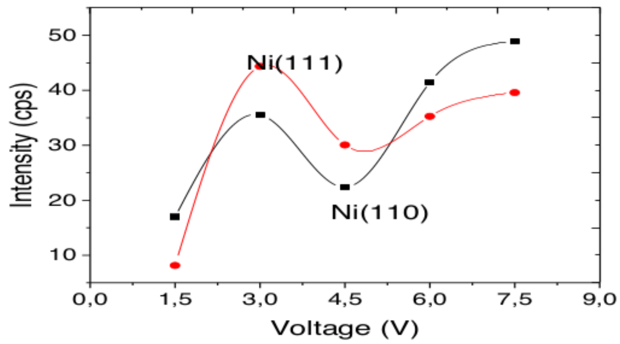

Figure 4 Effects of deposition voltage on diffraction peak intensity

The diffraction peak intensity reflects the regularity level of the crystal structure. The bigger intensity, the more regular the crystal structure. From Fig. 4, the deposition voltage influences the diffraction peak intensity. From the voltage 1.5 – 3 V the intensity increases, then decreases until the voltage of 4.5 V, then increases again. There are two directions of Ni crystal intensity observed those are direction [110] and [111]. At first, at the voltage of 1.5V Ni[110] intensity is higher than Ni[111], but at the voltage range of 3.0 to 4.5 V Ni[111] intensity becomes higher than Ni [110]. At the voltage over 4.5V intensity of Ni[110] is higher than Ni[111]. This shows that the voltages may influence in shifting the direction of the ordering of crystal structure that is regular at first then it becomes less regular and vice versa.

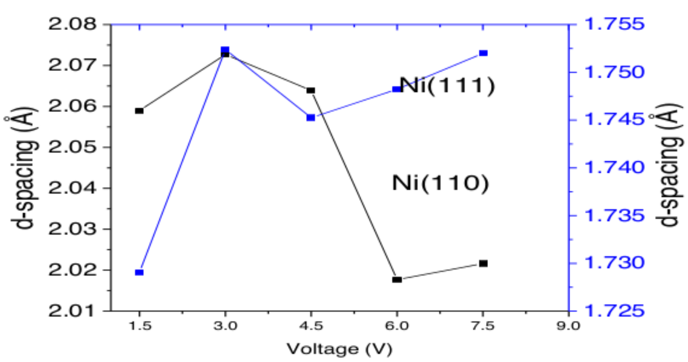

Figure 5 Effects of deposition voltage towards inter-planar distance

The *d*-spacing parameter is essential in the determination of resistivity due to its proportionality to the resistivity. The lower the *d*-spacing, the lower the resistivity and vice versa. Form Fig. 5, at first, *d*-spacing of Ni(110) and Ni(111) are equally low those are 2.06 Å and 1.73 Å. In line with the increasing voltage until 3 volt, both *d*-spacing get increased. However, both *d*-spacing get decreased for the deposition voltage of 4.5 volt. Specific condition was happened when the voltage is increased until 7.5 volt, that is *d*-spacing of Ni(110) gets decreased while d-spacing of Ni(111) gets increased. Thus, for the voltage over 4.5 volt the deposition can shorten the inter-planar distance on the direction of [110] while for the direction of [111] the increase of deposition voltage can increase the inter-planar distance of Ni crystal.

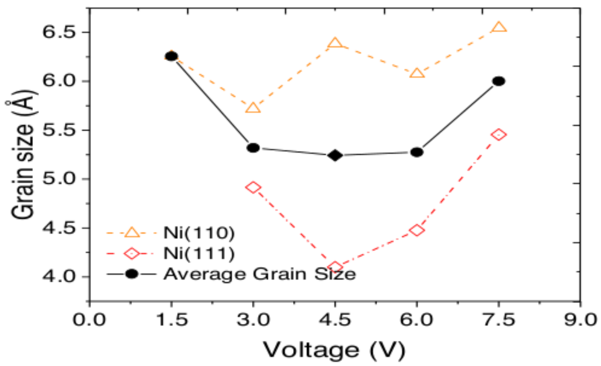

Figure 6 Effects of deposition voltage on grain size

The crystal grain size of Ni was determined with the Scherrer formula [22,23] on both directions those are [110] and [111], and for the result, the average value was taken. On the sample resulted from deposition on the voltage of 1.5 V, there is a diffraction peak on the direction of [110] but no on the direction of [111]. Therefore, on the direction of [111] the grain size cannot be defined or the film does not form as crystal structure. The increasing the deposition voltage until 4.5 V causes the grain size to get smaller from 6.26Å to be 5.24Å. It shows that the increase of the deposition voltage will increase of ion energy that can increase the mass transport of Ni. As consequence of this, the thickness of the Ni film increases as shown in Fig. 2 for the voltage 1.0 – 4.5 V. However, the increasing ion energy, the collision of Ni ion with Ni atoms which has formed on the Cu surface from deposition process before can disturb the crystal granules that were formed before, so the grain size gets decreased. When the voltage is increased until 7.0 V, Ni atoms can rejoin with the surface atoms so it produces new crystals with bigger size than the previous ones that is 6. 0Å.

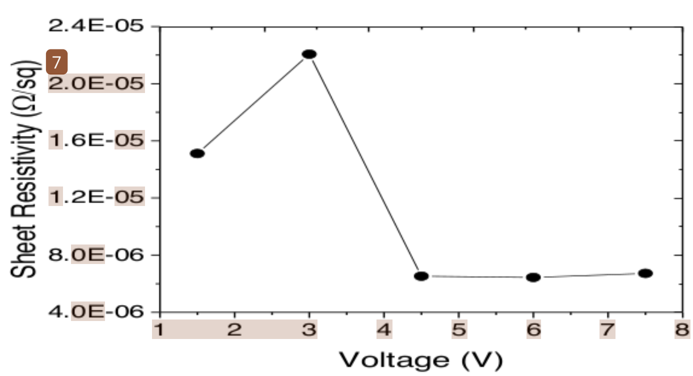

Figure 7 Sheet resistivity on the different deposition voltage

The relationship of the sheet resistivity of the Ni film towards the deposition voltages is shown in Fig. 7. At first, the Ni film from the deposition on the voltage of 3 volt has the sheet resistivity of 15.11 μΩ/sq. The factors that contribute to this value are the low intensity and the large inter-planar distance while those contribute to the decreasing the sheet resistivity is the large grain size even the largest of all samples that is 6.26 Å. Furthermore, on the deposition voltage of 3 V, Ni crystals are arranged regularly and may contributes to the low resistivity. However, this sample has the large even the largest inter-planar distance of all samples that is 2.07 Å, so the total resistivity of the Ni film becomes higher that is 22.06 μΩ/sq. On the deposition voltage from 6.0 V to 7.5 V the sheet resistivity is mostly contributed by the regularity of crystal structure and the low inter-planar distance particularly for Ni (110) plane that corresponds to the 2 theta angle of 44.18°. This sheet resistivity is approximately between 6.44 – 6.72 μΩ/sq.

4. CONCLUSIONS

Electroplating of the Ni to the Cu plate aided with the magnetic field parallel to the electric field on the deposition voltage variation has been successfully carried out. On the voltage from 1.5 – 4.5 V, the thickness of the film is proportional to the deposition voltages with maximum thickness of 5.78 μm while on the voltages over 4.5 V, the thickness of the film decreases to become 2.86 μm. The deposition voltage also produces the crystal structured Ni film with the dominant peak on the angles of approximately 44.18° and 52.38° that correspond to the directions of [110] and [111]. The diffraction peak intensity gets increased on the voltage of 3.0 V and 7.5 V. The deposition voltage variation also influences the *d*-spacing and the crystal grain size. On the voltage from 1.5 – 4.5 V the *d*-spacing profile of Ni[110] and Ni[111] are similar that is increasing until the peak on the voltage of 3.0 V then decreasing until the voltage of 4.5 V. On the voltages over 4.5 V, *d*-spacing of Ni[110] decreases while Ni[111] increases. Mean while, for the grain size on both ends of voltages those are 1.5 V and 7.5 V the grain size is high that is about 6.13Å, but between the two end voltages, the grain size decreases at about 5.28Å. All microstructure parameters influence the value of the sheet resistivity of Ni film. On the voltages over 4.5 V, the sheet resistivity decreases compared to other voltages. The biggest contribution on this value are the high level regularity of crystal structure, the low *d*-spacing of Ni[110], and the large grain size.

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