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by Oki Pak

Submission date: 23-Nov-2021 09:22AM (UTC+0700)

Submission ID: 1710732629

File name: Structure And Resistivity Of CuNi Thin Film Effects Of Electroplating Assisted With Parallel Magnetic Field On Deposition Time Variation.pdf (406.48K)

Word count: 3703

Character count: 18587

International Journal of Advanced Research in Engineering and Technology (IJARET)
Volume 11, Issue 10, October 2020, pp. 342-349, Article ID: IJARET_11_10_036
Available online at http://iaeme.com/Home/issue/IJARET?Volume=11&Issue=10
ISSN Print: 0976-6480 and ISSN Online: 0976-6499
DOI: 10.34218/IJARET.11.10.2020.036

STRUCTURE AND RESISTIVITY OF CU/NI THIN FILM –EFFECTS OF ELECTROPLATING ASSISTED WITH PARALLEL MAGNETIC FIELD ON DEPOSITION TIME VARIATION

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ABSTRACT

Cu/Ni thin film has been made with an electroplating method assisted with a parallel magnetic field on deposition time variation. The purpose of this research is to find out the thickness, microstructure, and resistivity of Cu/Ni thin film. The electroplating process was carried out with a magnetic field of 150 G, a solution temperature of 60°C, and an electrode distance of 4 cm. The solution used was made from the mixture of H_3BO_3 *40 gram,* $NiSO_4$ *260 gram,* $NiCl_2$ *60 gram, and* H_2O *1000 ml. The deposition time was varied from 60 s to 540 s. Deposition time has a positive influence on the thickness of the film, which is the longer deposition time the thicker Ni film. Deposition time also influences the microstructure parameter of Ni film. The Ni [111] diffraction peak is at an angle of approximately 44.8°. The intensity of the diffraction peak has an upward trend from the time of 60 s to 320 s and then it has a downward trend. Meanwhile, the profile of the grain size curve is similar to the distance of crystal plane which is from 60 s to 120s it goes down, then from 120 s to 420 s it goes up and then goes down again. However, the sheet resistivity has an upward trend towards deposition time.*

Key words: Cu/Ni thin film, electroplating, deposition time, parallel magnetic field

Cite this Article: Julius Saputra, Moh. Toifur, Okimustava, and Azmi Khusnani, Structure and Resistivity of CU/NI Thin Film – Effects of Electroplating Assisted with Parallel Magnetic Field on Deposition Time Variation, *International Journal of Advanced Research in Engineering and Technology,* 11(10), 2020, pp. 342-349.
http://iaeme.com/Home/issue/IJARET?Volume=11&Issue=10

1. INTRODUCTION

Cryogenic technology studies materials with very low-temperature under -150°C. Cryogenic freezing is carried out in an isolated freezer and happens continuously. In the food preservation with the cryogenic freezing method, the ice crystal formation will be much less compared to the conventional method, so the products do not experience much-added weight and the food quality is maintained [1]. Even more, cryogenic technology is used in a variety of fields, such as animal husbandry [2], and health [3-4]. The preservative media used is Liquid Nitrogen (LN2). LN2 has a freezing point of approximately -196,1°C until -198°C [5-6].

Along with the development of cryogenic technology, control towards temperature change becomes a critical factor. Therefore, it needs a low-temperature thermometer that can function well. One of the sensors that can be used is the Resistance temperature detector (RTD) based sensor. The materials for the RTD sensor is commonly made from metal or alloy, both in the forms of a coil or thin film [7-8]. The working principle of RTD is by using the resistance change influenced by temperature [9-10].

One of the metallic materials used in the manufacture of a thin film on RTD elements is platinum (Pt) because platinum has resistance towards oxidation, high accuracy, and good stability [11-12]. However, platinum is a relatively expensive metal. Therefore, to make a thin film on RTD, copper (Cu) and nickel (Ni) are used as alternatives [13-15]. Cu has the potential as a temperature sensor [16], but Cu is still less sensitive towards temperature change. This is because the resistivity of Cu tends to be still very low [17] and the characteristic of Cu is easily oxidized. Therefore, the Cu sensitivity can be increased by synthesizing it with Ni that has better resistivity to form the Cu/Ni thin film. Another advantage of Ni is that it has a better adhesion force compared to Pt, so it eases the film deposition [18]. Based on those reasons, Cu and Ni were chosen in this research. The synthesizing method used in this research is electroplating assisted with an external magnetic field accompanied variation on deposition time. Electroplating was chosen since it is economic, quick and the process is easily controlled [19-21]. The magnetic field is used to produce a magnetic induction force that will increase the homogeneity of ion distribution and increase substrate coating speed [22] and the formed film has a stronger texture [23]. This will influence the electrical characteristics of the material such as material resistivity [24].

2. MATERIALS & EXPERIMENTAL PROCEDURES

2.1 Substrate Preparation

The commercial copper was cut with a size of 1x3 cm2. The plate surface was cleaned with autosol metal polish, rinsed with distilled water, and continued with alcohol in an ultrasonic cleaner, then dried with a hairdryer. Next, a cutting sticker with a specific pattern was stuck on the copper surface and then copper outside the cutting sticker was dropped by using ferric chloride. The copper plate was lifted, and then cutting sticker was also taken off, cleaned with autosol metal polish, rinsed with distillation water. The copper surface was cleaned again with Pepsodent toothpaste, rinsed with distilled water, and rinsed with alcohol in an ultrasonic cleaner for about 10 minutes. Next, the sample was lifted, dried with a hairdryer, kept, and packed with tissue then put in a clip plastic and kept in the dry box [25].

2.2 Electroplating

In this stage, the solution consisting of H_3BO_3, $NiCl_2$, $NiSO_4$, and distilled water 1000 ml was prepared with stirring by using magnetism for 180 minutes. Before being plated, the substrate was weighed using the Ohaus-123 balance and the weight was noted as M_{Cu}. Next, it was installed as a cathode. A nickel plate was installed as an anode. The electroplating process was carried out on the voltage of 4.5 volt, electrode distance of 4 cm, one minute time, magnetic field intensity of 200 G, and a solution temperature of 60 °C. The deposition time was varied from one to ten minutes. After finishing, the sample was lifted and rinsed with distilled water and dried with a hairdryer. After dried, the sample was weighed and noted as $M_{Cu/Ni}$.

2.3 Data Analysis

The film thickness is calculated by using the equation [25-26] :

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

Where D is the thickness (cm^2), Δm is $m_{Cu} - m_{Cu/Ni}$ (g), ρ is the density of nickel (8.908 g/cm^3), A is the sample surface area (7.61 cm^2).

The analysis of the microstructure of Cu/Ni thin film was carried out by using XRD for angles of 2° to 100°. The diffraction angle, intensity, and FWHM were used to analyze the influence of deposition time on the change of intensity, grain size, and distance of the crystal plane.

The determination of sheet resistivity was carried out by using a homemade four-point probe by varying current rates (I) and (V). After getting the data set of (I_i, V_i) then being plotted and fitted based on the linear equation $y = ax+b$. The sheet resistivity can be calculated by using the slope of graph a, in accordance with:

$$R_s = \frac{\pi}{\ln 2}\frac{V}{I} = \frac{\pi}{\ln 2}a \quad (2)$$

With, R_s is sheet resistivity (Ω/sq), V is voltage (V), and I is current (ampere). Next, the influence of deposition time on the microstructure was used to analyze the value of R_s on the deposition time variation. It is possible that several distractions occur on the sample such as the impurities and defect on the crystal will influence the value of R_s so the relationship of R_s towards deposition time does not apply ideally.

3. RESULTS AND DISCUSSION

The electroplating process was carried out and has produced nickel film on a copper plate to becomes Cu/Ni. The thickness of Ni film on a variety of deposition time is calculated by using equation (1). The results were shown in table 1.

Table 1 The relationship between time deposition and film thickness

Deposition time, *t* (s)	**Thickness, *D* (μm)**
60	6.02
180	8.12
300	8.99
420	9.46
540	11.70

The thickness is approximately ten times thicker than that produced without the magnetic field [13, 22]. Here, the use of a magnetic field has accelerated the mass transport process of Ni ion in the solution close to the anode to stick on the Cu surface. This is suitable to Jia and Wu [27] revealed that the amount of particle in the process of deposition assisted with a parallel magnetic field is larger than that without it.

Next, figure 1 shows the diffract gram of XRD of the Cu/Ni sample. The crystal structured has been exhibited by all samples. The dominant peaks of Ni diffraction are located at 2 theta angle of approximately 44.8° and 52.2° with hkl plane direction [111], and [002].

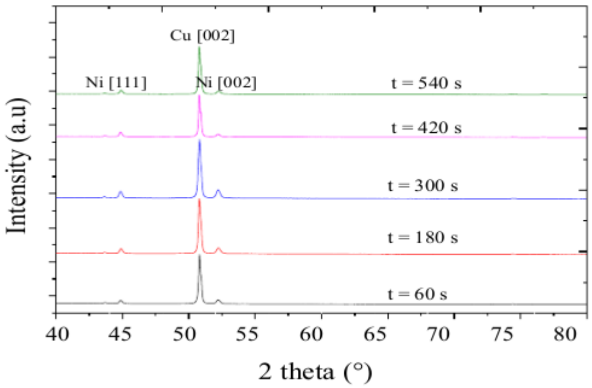

Figure 1 Diffractogram of Cu/Ni substrate on deposition time variation

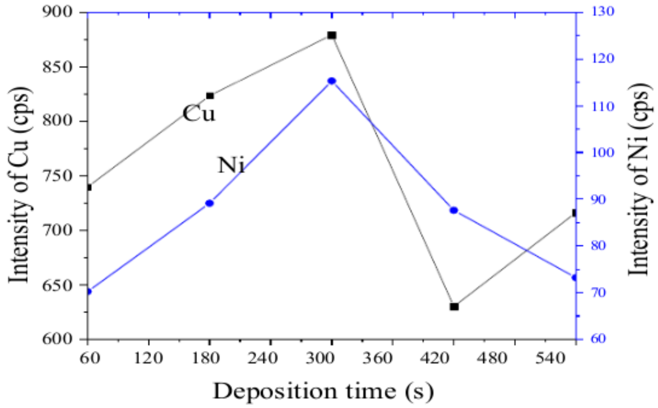

Figure 2 The Graph of the relationship between time deposition and intensity

Fig 2 shows the graph of the influence of deposition time on the diffraction intensity of Cu and Ni. The curve shape of diffraction intensity on Cu and Ni materials tends to be similar, so it shows the crystal structure of Cu influences the crystal structure of Ni that is superimposed on the Cu surface. The highest peak of Ni diffraction intensity of both phases occurs at a time of 300 s that is 115.30 cps, while the lowest intensity occurs at deposition time of 60 s that is 70.22 cps. It occurs that from the deposition time of 60 s to 300 s the crystal structure regularity level gets up. However, the additional deposition time until 540s causes the crystal structure regularity level of the film gets down. This is influenced by the hydrogen as mentioned above, so it obstructs the coating process. In that condition, the existence of an external magnetic field does not support the Ni atomic arrangement on the Cu surface.

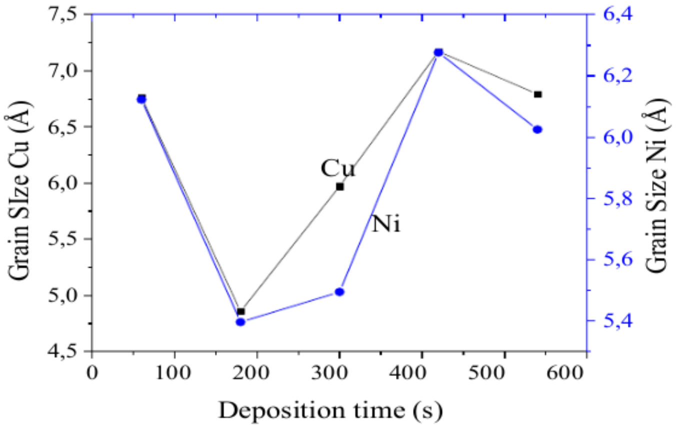

Figure 3 Graph of the relationship between time deposition and grain size

On the Ni deposition assisted with the magnetic field, deposition time influences the grain size of Ni crystal formed at the direction of [111] (figure 3). Firstly, the grain size from the deposition time of 60 s to 180 s decreases then from 180s to 420 s increases and then decreases again. The range of grain size is approximately 5.40 Å to 6.30 Å. The smallest Ni grain size is founded on the sample of resulted from deposition at 180 s, while the largest is from deposition at 420 s. In the fluctuation of grain size, it is estimated that the magnetic field plays a role in guiding the Ni ferromagnetic granule growth in the same direction with the magnetic field [28]. This goes along with the result of research by Zhou et al [29-30] that have conducted Fe/Si plating assisted with a parallel magnetic field. The film surface of Fe/Si becomes surging and clear stripped.

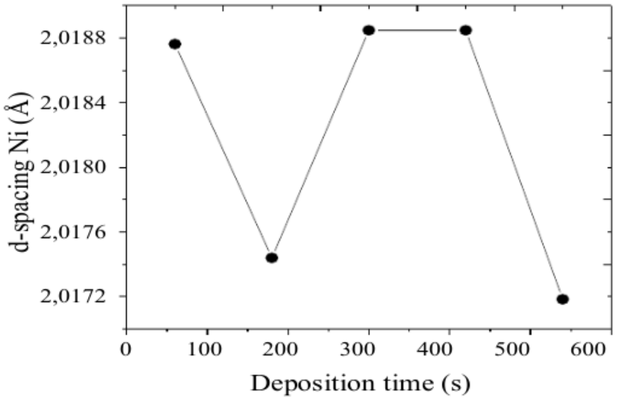

Figure 4 Relationship between deposition time and d-spacing

Deposition time together with the external magnetic field indirectly influences the distance of crystal planes. First, the distance of the crystal plane goes down, then up then down again. Here, the magnetic field plays a role to develop Ni crystal formation with a particular size. The combination of the magnetic field force field on Ni with electrical force and magnetic driving force near the cathode surface makes the distance between Ni planes changing towards the length of deposition.

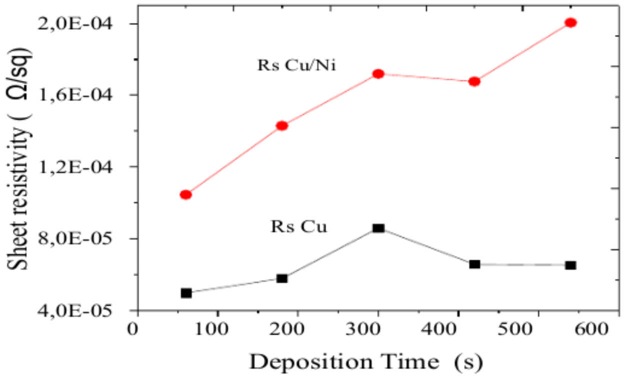

Figure 5 Relationship between deposition time and sheet resistivity

Deposition time has a great impact on the sheet resistivity of Cu/Ni film. Figure 5 shows that the sheet resistivity of Cu film before being coated by Ni is in the range 4.96×10^{-5} ohm/sq to 6.51×10^{-5} ohm/sq. However, after coated by Ni, the sheet resistivity of Cu/Ni increases quite significantly more than twice from that before, which is in the range of 10.44×10^{-5} to 20.05×10^{-5} ohm/sq [31]. It is associated with the diffusion process of Ni to Cu atoms for producing an alloy at the interface. The diffusion is quite likely to happen due to the similarity of the atomic radius of Ni and Cu, which are 1.49 Å and 1.45 Å respectively. The largest resistivity is associated with a sample from deposition during 540s that is 20.05×10^{-5} ohm/sq.

The value of the R_s depends on the value of the microstructure parameters of the Ni film which consists of the regularity level of crystal structure, the distance between the crystal planes, and the grain size. In addition, the deposition time also determines the thickness of the film. In my previous studies [32], the longer the deposition took the smaller the R_s, but here it is getting higher. Hence the magnetic field during deposition plays a significant role in increasing this R_s value, as was appointed by Yundan [28] concerning the presence of granules parallel to the direction of the external magnetic field. Also as the impact of the use of a parallel magnetic field, the film surface becomes surging and clear stripped [29-30].

4. CONCLUSIONS

In this electroplating assisted with the parallel magnetic field, deposition time can give a change to the thickness of the film and microstructure parameters such as the intensity of the diffraction peak, crystal plane distance, and grain size. Deposition time variation can increase the sheet resistivity of Cu/Ni film. With a deposition time of 60-540 s, the sheet resistivity can increase 2.5 times. The higher Rs is due to the use of a magnetic field in the deposition process which causes the granules to appear in the direction of the magnetic field and the surface becomes wavy and clear stripped. The granule content and strip are proportional to the deposition time.

ACKNOWLEDGMENT

The researcher expresses his deepest gratitude to the Ministry of Research and Technology/ National Research and Innovation Agency which has provided research funding through the 2020/2019 through Post Graduate Research Grant Scheme with a contract number of NOMOR: PTM-030/SKPP.TT/LPPM UAD/VI/2020.

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