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Fakultas Matematika dan Ilmu Pengetahuan Alam
(Faculty of Mathematics and Natural Sciences)
Universitas Ahmad Dahlan**

Proceeding of The International Seminar on Natural Sciences and Applied Natural Sciences

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DEPOSITION OF Cu/Ag FILM AT THE VARIOUS DEPOSITION TIME FOR RESULTING THE HIGH QUALITY DECORATIVE SILVER

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ABSTRACT

Deposition of Cu/Ag film with electroplating method at the various deposition time of Ag has been performed to produce high quality decorative silver. The criteria of high quality performance consist of two parameters, i.e. the ordering of crystalline structure and the reflectance coefficient.

Copper (Cu) plate was used as substrate which is coated with silver (Ag) in the KCl plating bath with various deposition times from 5 up to 8 minutes. The ordering of crystalline structure was characterized by XRD, while reflectance coefficient is obtained with measuring the reflected polychromatic rays (lamp 10 volt, 1.2 mA) by LDR.

The result shows that Ag film 7 minutes deposition has the highest ordering of crystalline structure, and also this film has the reflectance coefficient of about 85%. Thereby, this film is the best quality for decorative silver.

A. Introduction

Gold, copper and silver is materials that most found in society as household instrument, decorative and accessories. For this impotancy, the appearance is significant by considerate apart of decorative function is to tend to beautiful color.

Since the raising of the basic material price, industry that produce the household instrument become paralyze and power of society for buying these products be decrease. As published in Kompas (Anonym, 2004) the about 95% from 340 metal industry in Ceper, central Java stop production.

Electroplating is one of alternative method to overcome these problems. Electroplating is process of thin film coating on the surface of metal with other ones (Eckertova, 1986; Ohring, 1992). The maximum thick of film is several hundred microns. In this process, basic material that coated has not too the expensive material, as: zinc, copper and steel. Because the coating material is too thin, the production cost can be reduced significantly, while this performance is still restrained. This is quite probable to increasing the price. By comparing with others techniques, known as physics deposition as sputtering and

evaporation, the cost for operating this technique is so cheap (Wasa and Hayakawa, 1992; Konuma, 1992). Therefore, electroplating is quite efficient, able to produce the thin film even ultrathin film faster than other methods can be operated to the area of surface wider than others, able to produce the film with high ordering crystalline structure.

In this paper we will report the results of optimization of electroplating machine on the making of Cu/Ag film at the various time depositions. The observed parameter is the ordering of crystalline structure and its reflectance.

B. Experiment Method

1. Material

The materials used in this research including: copper plate as substrate, silver plate as coating material with size of 3 cm \times 1 cm \times 0,04 cm and Kalium Klorida (KCl, potassium) as plating bath.

2. Instrument

The instrument used in this research including: ultrasonic cleaner 60 Hz for washing the surface sample, electroplating machine completed with regulator for electrode distance, electrode voltage, and solution temperature controller. Instrument for characterizations the sample include: XRD spektrometer for observed

microstructural of Cu/Ag film, four point probe for obtaining resistivity of sample, LDR (light diode resistance) is used to obtain the appearance level of sample. The light source is polychromatic rays from the lamp 12 volt, 1.2 mA.

C. Procedure of Research

1. Preparing sample

Process of preparing sample include: Cutting the copper (Cu) plate with size of 2.5 cm \times 1 cm \times 0.002 cm. Then refining the surface of Cu with abrasive paper number 300, 900 and finished with brasso. The next step is washing the samples with *ultrasonic cleaner* during 10 minutes at the temperature of 40° C and wasing with destiled water after at the time of 10 minutes and finished with alcohol 95%, at the time of 10 minutes.

2. Deposition process

Deposition process is done with following procedure:

- a. Filling the bath with plating bath of potassium with concentration of 10 gram/liter
- b. Put Cu plate on the cathode and Ag on the anode
- c. Connecting the two electrodes on the DC 5 volt power supply.
- d. Heating the solution until temperature of 90°C

- e. Operating deposition process in the various of time deposition that are 5, 6, 7, and 8 minutes. This process divided on two stages, the first stage deposition during 3 minutes, and the second stage is deposition during 2, 3, 4, and 5 minutes.

D. Results and Discussion

At the part below, it is conducted the results including XRD spectrums for Cu substrate and Ag film at the various time deposition, sheet resistivity of Cu/Ag and reflectance of Cu/Ag.

1. XRD spectrum of Cu substrate

Before we analyze the structure of Ag film, it is important to analyze the XRD spectrum of Cu substrate, due to: (1) make obtaining the diffraction peaks of Ag XRD spectrum easier without notice the Ag XRD spectrums from data sheet. The Ag peaks are the peaks which different from the Cu peaks. (2) The ordering of Cu crystalline influence to the ordering of crystalline structure of Ag, primary at the interface of Cu and Ag films. It is due to the difference of lattice constant of two elements. In Fig. 1 showed the XRD spectrum of Cu substrate. The existence of diffraction peaks show that Cu has crystalline (or polycrystalline) type.

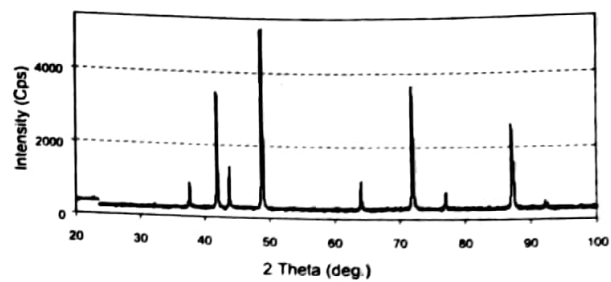


Fig. 1 XRD spectrum of Cu substrate

The peaks of spectrum are held in the angles of 37, 74, 42, 43.90, 48.90, 64.08, 71.96, 77.06 and 87.50°. Intensity of spectrum shows the ordering of crystalline structure. The higher of intensity the more order of crystalline. The highest intensity is 5354 cps that according to the angle of 48.9°. The index Miller is [111].

2. XRD spectrum of Ag film at the various of Ag deposition time

In Fig.2 showed the X-ray spectrum from Ag film resulted from deposition at time 5, 6, 7 and 8 minutes. As shown in Fig. 2, it is founded the adding peaks in the around of angle of 34°. This peak according to the PCDF table is Ag peak.

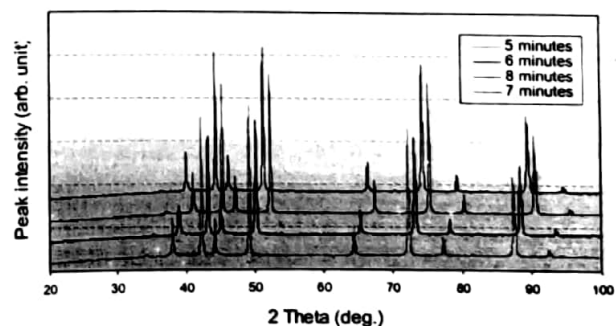


Fig. 2 XRD spectrum from Cu/Ag film on the various deposition time of Ag

Quantitatively, the diffraction angles and intensity of Ag and Cu as tabulated below.

Table 1 Diffraction angles and intensity of Ag and Cu (Cullity, 1978)

Time (minutes)	Ag		Cu	
	angle	Intensity	angle	Intensity
5	34.35	3526	49.3	38386
6	34.25	3290	49.25	29890
7	34.40	3572	49.35	35650
8	34.40	3432	49.35	36680

If we notice the diffraction angles as showed in column 2 and 4 table 1, it is appear that the position of peaks and intensities are slightly fluctuate up on variation of deposition time. For analyze data more easy, in fig. 3 displayed graph of intensity of Cu and Ag up on deposition time.

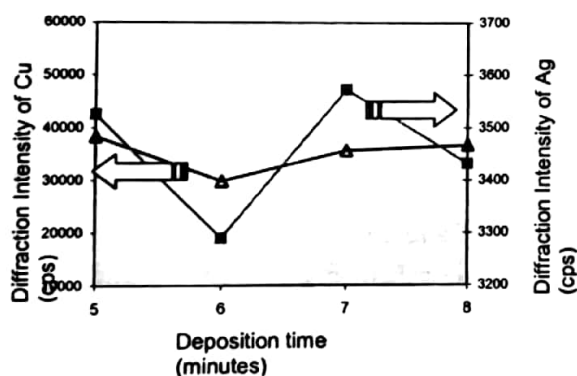


Fig. 3 Graph of diffraction intensity of Cu and Ag at various time deposition

From figure, it is appear that the highest peak for Ag is according to sample deposited during 7 minutes, that is 3572 cps. Really, the highest crystalline structure of Cu is according to the 5 minutes deposition

time, that is 38386 cps, while intensity of Cu on the highest intensity of Ag is 35650 cps. Here, the highest intensity of Ag not automatically is according to the highest intensity of Cu. It is due to mismatch of lattice constant (a) between Cu and Ag, and the difference of atomic size between them. The lattice constant of Cu and Ag are 0.36147 nm and 0.40856 nm (Smith, 1990) respectively. Because the lattice constant represents the distance between two atoms, the distance between 2 Ag atoms is longer than it between 2 Cu atoms. The difference of lattice constant between Ag and Cu is 0.04709 nm. The size of Cu atom (from the atomic radius) is 0.128 nm shorter than atomic size of Ag that is 0.144 nm, even though Ag in the ionic form has radius shorter than it atomic radius that is 0.113 nm.

So, the regularity of Cu crystalline structure depends on the regularity of Ag crystalline. The more regular of crystalline structure of Ag the more irregular the crystalline structure of Cu. On the contrary, in the circumstance that crystalline structure of Cu relatively is more order as that resulted at the deposition time of 5 and 8 minutes, the crystalline structure of Ag is less order. As the mentioned above, here Ag atoms are compelled have suitable arrangement with Cu atoms whereas the lattice constant oh the two atoms is not same. There fore if crystalline structure of Cu atoms arises, the

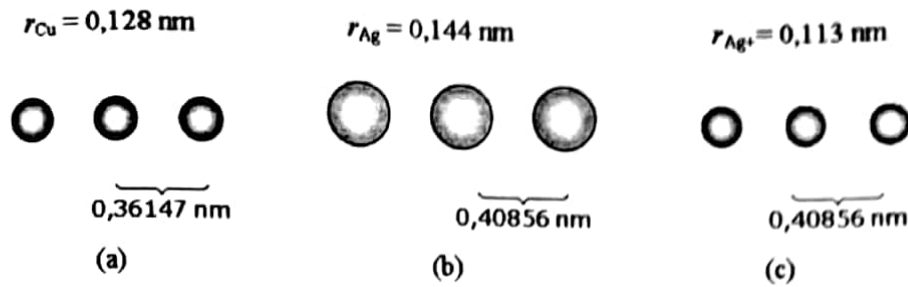


Figure 4 Description of size and lattice constant of Cu and Ag.

ordering crystalline structure of Ag decrease. From the results it is known that the optimum time for achieving Ag film that have the highest ordering crystalline structure is 7 minutes.

3. Sheet Resistivity of Cu/Ag films at the various time depositions

In Fig. 5 displayed curve of sheet resistivity (R_s) of Cu/Ag film as function of Ag deposition time. By assuming that sheet resistivity of Cu is constant, so the change of sheet resistivity at the various deposition times is influenced only by Ag film.

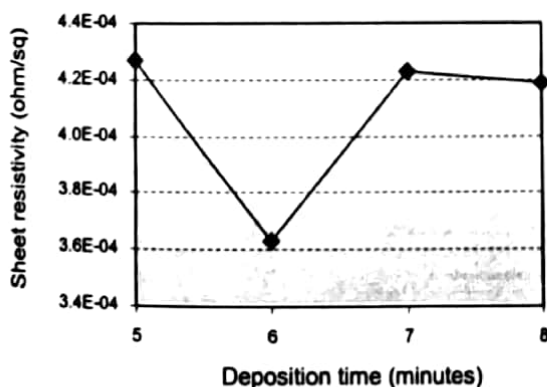


Fig. 5 Sheet resistivity of Cu/Ag as function of deposition time of Ag

The R_s value is around $4.2 \times 10^{-4} \text{ ohm/sq}$, except for sample resulted on the time deposition of 6 minutes, which have minimum value of R_s , $3.6 \times 10^{-4} \text{ ohm/sq}$.

4. The Reflectance of Ag film at the various time depositions

In Fig. 6 showed reflectance Ag film up on polychromatic ray from lamp with specification 12 V, 1.2 mA. From the result show that Ag film resulted by deposition on during 7 minutes has the highest reflectance (R), that is 94.20%. In this case, the ordering of crystalline structure has big role for obtaining R . As explained before that film produced in that time deposition (7 minutes) has the most order crystalline structure. Therefore the R is the highest too. The minimum R of Ag film concerning with time deposition of 8 minutes, that is 76.60%. It is due to saturation solution of KCl as the consequence from inequilibrium the number

of electron tends to anode and the number of Ag ion tend to cathode.

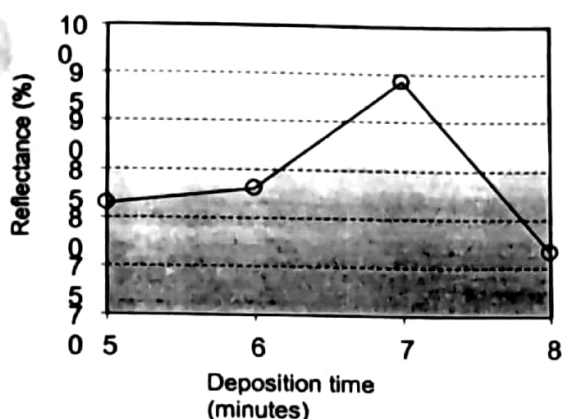


Fig. 6 Reflectance of Ag film as function of time deposition

Because the mass of Ag ion is heavier than electron mass, the moving of Ag ion is quite slowdown compared with the moving of electron. As consequent, accumulation of electron will be held at the area around of anode. It accumulation of electron will prevent the ionization process of Ag (anode) and deposition process will be disturbed. Because the number of Ag ion deposit is little, cathode surface become dirty and the R tend to low.

E. Conclusion

From above discussion, we conclude some points as revealed below:

1. The time deposition influence to the order of crystalline structure of film and reflectance coefficient.
2. The Ag film resulted in the time deposition of 7 minutes is the most

suitable sample used as decorative silver material indicated by the high ordering of crystalline structure and the high reflectance in responding the polychromatic rays.

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