Influence of Annealing Temperature on Surface Morphological and Electrical Properties of Aluminum Thin Film on Glass Substrate by Vacuum Thermal Evaporator

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Abstract. This paper explains the effects of the annealing temperature on structural and electrical properties of Aluminum (Al) thin films. Al thin films were deposited on glass substrate by thermal vacuum evaporator. The films were then annealed at 100°, 200°, 300°, 400°, and 500°C for 1 hour. The surface morphology of Al films after annealing were characterized using atomic force microscope (AFM) and field emission scanning electron microscope (FESEM). The electrical properties were characterized using four point probe. From the results of this experiment, the roughness of Al films gradually decrease from 8.5 nm (before annealing) to 7.7 nm and the grain size gradually increase from 127 nm to 145 nm, when the temperature of annealing increased. The resistivity of the films was also decreased from 2.32 x10⁻⁵ ohm.cm to 1.9 x10⁻⁵ ohm.cm when the samples were annealed from 100°C to <400°C that depended on roughness. However, when annealed from 400°C to 500°C, the resistivity shows dependency on grain size, which result on the increasing of resistivity to 2.77 x10⁻⁵ ohm.cm.

1. Introduction

Aluminum (Al) is widely used in many application such as aircraft, beverage cans, thin film IC interconnect, thin film transistors, solar energy, flat panel displays, optical mirrors, and many more [1–3]. This is due to the excellent properties of aluminum such as light weight, good reflectance, good adherence to glass, good mechanical properties, corrosion resistance and easy to recycle. In addition, Al is the most abundant metal in the earth’s crust and the third most abundant element. Physically, Al is a metallic compound that has silvery white in color and low melting point of 600°C compared to silver of 961°C and copper of 1085°C.

There are many techniques used to deposit Al on a substrate, like sputtering, electron beam evaporation, thermal evaporation, and plasma-assisted atomic layer deposition [4–9]. Compare to others physical vapor deposition (PVD) technique such as sputtering, ion plating, and ion beam sputtering, thermal vacuum evaporation is very suitable to deposit Al, since it was easy set up and cost effective [7].
In this study, we present the effect of thermal annealing on the electrical and structural properties of Al thin films deposited on glass by thermal vacuum evaporation. One of the methods that can enhance the properties of thin film is post-thermal treatment. Both electrical and structural properties of thin films will change due to the segregation and realignment of crystal atoms. There are many applications on microelectronics and nanotechnology that require low resistivity and smooth surface of thin films. Thus, by performing annealing on Al thin films, the grain size became larger as well as smoothing the surface. Moreover, it was also decreasing the resistivity of the films.

2. Experimental Method
The experiment begun by preparing glass substrates and high purity of aluminum wire (99.99%). Firstly, the substrates were ultrasonically cleaned with acetone and ethanol for 10 minutes each respectively, then rinsed with deionized water (DI water) to remove contamination from the surface of the substrates. Then the substrates were dried by blowing pure nitrogen gas over them before deposition.

Thermal vacuum evaporator (Ulvac Kiko VPC-061) was used to deposit Al film on the substrates. Al target was placed inside tungsten boat, and the deposition process was conducted in vacuum condition with a base pressure of ~10^{-3} Pa. The current supply was turned on and increased gradually from 0 until 35 Ampere, which results in ample heat to melt the Al. Al was vaporized after the temperature achieved ≥600°C. The deposition time was set for 2 minutes.

After all films successfully deposited, annealing process ready to applied on. The annealing process was conducted in an electric furnace in temperature ranging from 100°C-500°C for 1 hour. One of the sample that was not applied to any heat treatment in which called as deposited.

The samples were characterized using Four Point Probe (Model: Lucas Labs Pro4) to measure the electrical properties and the resistivity were measured and averaged from five areas on the same substrate. The surface properties of the samples were then characterized by Atomic Force Microscope (AFM) system (Model: XE-100 Park Series) and Field Emission-Scanning Electron Microscope (SEM) FE-SEM (Model: JEOL JSM-7600F) to measure the roughness and the grain size of the films.

| Process parameter       | Conditions                                     |
|-------------------------|------------------------------------------------|
| Base Pressure           | 10^{-3} Pa                                     |
| Target                  | Aluminium wire: 99.99% purity mass: 35 mg      |
| Target-substrate distance| 10.5 mm                                        |
| Boat                    | Tungsten                                       |
| Substrate               | Glass                                          |
| Substrate temperature   | Room temperature                               |
| Current                 | 35 Ampere                                      |

3. Results and Discussion
Figure 1 shows the FESEM images of Al films before and after annealed at various temperatures ranging from 100°C, 200°C, 300°C, 400°C, and 500°C for 1 hour. It is clearly seen that the grain size of the Al films has a good uniformity and no significant change after annealing treatment. Generally, all films indicate uniform structure and most of the particles have nearly similar sizes. Based on the
images, the particle size is around 130 nm. Moreover, there were no crack occurred on Al surface morphology even when annealed in temperature near the melting point of Al.

![Figure 1. FESEM image of the Al films (a) as deposited, and post-annealed at (b) 100°C, (c) 200°C, (d) 300°C, (e) 400°C, and (f) 500°C](image)

Figure 2 shows the AFM images of Al films on glass with different annealing temperature. It is clearly seen that all films deposited by thermal vacuum evaporator have a tendency to result in smooth surface. The grains are relatively large and regular in shape with clear definition between the grains. Based on the images, the roughness of the films are around 8 nm. It is showed a clear evidence of hillocks protruding 20 to 40 nm from the film surface. This is evidence of the aluminum being deposited in a condition of compressive stress.
Figure 2. AFM image of the Al films (a) as deposited, and post-annealed at (b) 100, (c) 200, (d) 300, (e) 400, and (f) 500°C

Figure 3 shows the inversely graphs of grain size and roughness of Al films before and after annealing treatment. Increasing the annealing temperature of the films resulted in increasing the grain size due to the grains partially melt and then rearrange again. This makes the grain size become larger than the original. Moreover, the roughness of the films were decreased, which means indicates improving the film quality due to the smoothness of the films surface.
Figure 3. Grain size and roughness of the Al films as function of different annealing temperature

Figure 4 showed the resistivity of Al films before and after annealed at different temperature. After annealing, the resistivity of the Al films decreased reach a minimum from $2.64 \times 10^{-5} \, \Omega \cdot \text{cm}$ to $2.32 \times 10^{-5}$, $2.18 \times 10^{-5}$, and $1.9 \times 10^{-5} \, \Omega \cdot \text{cm}$ when annealed at temperatures of 100°, 200°, and 300°C respectively. This is believed due to the atoms structure of Al toward an orderly arrangement from the initial state and the grain growth, increase in grain size which reduce the grain boundary scattering of charge carriers. However, the resistivity of Al films sharply increases at a certain point from 400° to 500°C. These can be attributed to a void growth phase of agglomeration, where the total electrical resistivity spiky increases with further increasing in temperature.

Figure 4. The resistivity of the Al films with different annealing temperature
4. Conclusion
From this experiment it was observed that deposition Al films on glass substrates by thermal vacuum evaporator result in smooth surface due to the directional line-of-sight deposition nature of thermal evaporation technique and clearly uniform. The annealing process resulted in changes the surface morphological of the films, increasing the grain size and decreasing the roughness. Moreover, When the films annealed from 100 to <400°C, the resistivity of the films depended on roughness. However, when annealed from 400 to 500°C, the resistivity shows dependency on grain size. Therefore, we can conclude the optimum annealing temperature of the Al thin films in this experiment was in around 350°C.

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