# Effects of annealing in air on structure and properties of Al films

## prepared by thermal evaporation

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Abstract. Al films were prepared on quartz substrates by thermal evaporation. The effects of annealing in air on structure and optical and electrical properties have been studied. It is found that the annealing in air will affect on structure and morphology of the films, which results in the difference in the optical and electrical properties. The as-deposited film is amorphous, the films annealed at 200 and 400°C are polycrystalline. After annealed at 600°C, the film was oxidized and changed to porous  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>. The film annealed at 200 °C has the maximum reflectance and at 400 °C has the minimum resistivity in all samples. While for the film annealed at 600 °C, the resistivity is close to be infinite, the reflectance is the minimum at wavelength ranging from 400 to 800 nm in all the samples.

#### Introduction



In previous investigations, it is found that the deposition and annealing temperature will effect on the structure, surface morphology, electrical and optical properties of metal films[8].For Al films, For example, C.A. Volkert and C. Lingk investigated Effect of compression on grain growth in Al films[9].

In this work, we studied the effects of annealing in air on structure and optical and electrical properties of Al films. It is found that the annealing in air will affect structure and morphology of the films, which results in the difference in the optical and electrical properties.

#### **Experimental details.**

Al films were deposited on quartz substrates by thermal evaporation under identical conditions using Al (99.99% purity) strips. Electric current for evaporation was 70 to 75 ampere and deposition pressure was  $5.0 \times 10^{-3}$  Pa. The deposition rate and thickness (about 70nm) were measured with a



quartz crystal micro balance. The surface morphology of the samples was examined by <u>atomic force</u> microscopy (AFM) (CSPM 4000). The average surface grain size (ASGS) and the root mean square (RMS) surface roughness of the films were calculated from AFM images. The reflectance and transmittance were measured in the  $200 < \lambda < 800$  nm wavelength range by use of a double beam spectrophotometer (UV-2450). Electrical resistivity was measured by a four-point probe method.

## **Results and discussion.**

## 2.1 The crystal structure and surface morphology.

Figure 1 presents XRD patterns of the samples. The as-deposited Al film is amorphous because of not peaks in XRD patterns in Fig. 1(a). However, for the annealed Al films, there are many broad peaks can be seen in Fig. 1(b-d). As shown in Fig. 1(b-c), there are broad diffraction peaks at (111) and (200) in the XRD pattern of the film annealed at 200 and 400°C. Comparing Fig. 1(b) with Fig. 1(c), it is found that the peaks show higher and stronger as the increase of the annealing temperature, and a new peak at (311) appears in the film annealed at 400°C. Additionally, there is no indication of the oxidization of Al film, which shows that the crystallization not oxidization is mainly below 400°C in air. This is similar to Ag films annealing in air[8]. But, when the annealing temperature further increases to 600°C, we can find the evident indication of the oxidization of Al film in Fig. 1(d). Comparing the diffraction peaks position with the data from JCPDS international diffraction data base, we find the peaks corresponding to the (111), (200), (311), (222), (400), (511) and (400) plan of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> with hexagonal structure[10].



Fig. 1. XRD patterns of Al films:(a) as-deposited; annealed in air for 1h at (b) 200°C, (c) 400°C, and (d) 600°C.



Fig.2. AFM images of Al films: (a) as-deposited, annealed at (b) 200 °C, (c) 400 °C and (d) 600 °C.

The evolution of surface morphology of the samples is presented in Fig.2. For the as-deposited Al film, the grains are spherical homogeneous with clear-cut, RMS surface roughness and SAGS are up to 1.32 and 65.2nm, respectively. While for the annealed film, comparing Figs. 2(a) and Figs. 2(b-c), we can find that, the surface grains become even tighter, RMS roughness and ASGZ change

to 1.27, 1.96 nm and 68, 82 nm when the annealing temperature increases to 200 and 400°C, respectively. And there are some voids with diameter ranging from 350 to 550 nm are visible in Figs. 2(c), which derives probably from the internal bubble, defect, stress and impurity during preparation process. However, when the annealing temperature increases to 600°C, the surface fills entirely with the voids. These voids are porous  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> proved in Figs. 1, not different from the voids annealed at 400°C. And the diameter range from 1000 to 1200 nm, the RMS and SAGS increase to the maximum, 2.57nm and 107nm, respectively.

## 2.2 Optical and electrical properties.



Fig.3. Reflectance at 600 nm wavelength and RMS roughness of samples as a function of annealing temperature.



Fig.4. Reflectance and resistivities of the Al films as-deposited and annealed: (a) Reflectance, (b) Resistivities.

Fig. 4 shows reflectance and resistivities of the as-deposited and annealed Al films. Reflectance at 600 nm optical wavelength of the films as a function of annealing temperature is also presented in Fig. 3. It is clear that the oxidization and RMS roughness affect directly on the optical reflectance, As indicated in Fig. 3 and 4(a), because of the oxidization and maximum RMS roughness, the reflectance of the film annealed at 600 °C is the minimum. While the films annealed at lower than 600 °C have higher than 75% reflectance at wavelength ranging from 400 to 800 nm, film annealed at 200 °C has the maximum reflectance because it has the minimum RMS roughness. Resistivity, as

presented in Fig. 3(b), decreases linearly to  $16.0\pm3.5 \ \mu\Omega$  at 400 °C annealed, But after Al film is annealed at 600 °C in air for 1 h, the resistivity is close to be infinite because of being oxidized into  $\gamma$ -Al<sub>2</sub>O<sub>3.</sub>

## Conclusion

Al films with 70 nm thickness were prepared on quartz substrates by thermal evaporation technique, and subsequently annealed in air for 1h at temperature ranging from 200 to 600°C. The effects of annealing in air on structure, the electrical and optical properties of Al films have been studied. For the films except annealed at 600 °C, the structure changes from noncrystalline the as-deposited to crystalline the annealed, reflectance is have higher than 75% at wavelength ranging from 350 to 800 nm, the minimum is at 200 °C, resistivity decreases linearly and the minimum is at 400°C. While the film annealed at 600°C was oxidized and changed to  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, the resistivity is close to be infinite, the reflectance is the minimum. So, our results show that the annealing in air also can improve the electrical and optical properties of Al films, and the bubbles, defects, impurities, and internal stress will affect the oxidized annealing temperature.

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