Thermal Stability of Au Thin Film Deposited on Al₂O₃ Substrate with RuO₂ Adhesion Layer

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RuO₂ を接合層に用いた Al₂O₃ 基板上の Au 薄膜の熱安定性

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The effect of RuO₂ thin films on stabilizing the thermal properties of Au thin films deposited on Al₂O₃ substrates was investigated. Dependencies on temperature of both sheet resistance and adhesion strength were measured. The adhesion strength of the Au/RuO₂ system was constant with temperature, while that of the Au/Pt/Ti system decreased as increasing temperature beyond 400°C. The adhesive properties of the Au/RuO₂ system were not influenced by heating and stable up to 800°C. The sheet resistance of the Au/RuO₂ system remained constant up to 700°C. The RuO₂ thin films can operate as heat-resisting adhesion layers in Au thin films. Au thin films were highly oriented in the Au(111) plane.

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1. Introduction

Recently, Au thin films are widely used as electrodes fabricated on the ceramics substrates. In general, Au thin films do not have good adhesive properties with Al₂O₃ substrates. For the purpose of improving the adhesive properties, reactive metals such as Ti, Zr and W were inserted as adhesion or glue layers.¹⁾ But these metals diffuse and react with Au films above 400°C.¹⁾ These phenomena deteriorate the adhesive properties and the resistance of Au films.

In order to prevent the diffusion of reactive metals, diffusion barriers²⁾ such as Pt, Pd, TiN and TiW films were inserted between Au thin films and adhesion layers. However, the components of these layers also diffuse into Au thin films heated at about 600°C. Furthermore, the components of the substrates diffuse into Au thin films through diffusion barriers at the same temperature.

Therefore, it is necessary to improve thermal stability of the adhesion or glue layers. Instead of reactive metals, several oxide thin films such as Al₂O₃, ZrO₂, SiO₂, TiO₂, SnO₂, WO₃ and RuO₂ were tried to use as the inserted layers between the Au thin films and the substrates. Within these oxide thin films, RuO₂ films were the most promising adhesion or glue layers. The purpose of this study is to stabilize the Au thin films on the RuO₂ inserted layers during heating.

2. Experimental

2.1 Film deposition

Commercial Al₂O₃ plates (purity > 99.5%) were used as the substrates for thin films deposition. As-received Al₂O₃ substrates were polished and lapped to 0.6 mm thick. The lapped average surface roughness (R_a) was 50 nm.

The sputtering method was used to deposit RuO₂ thin films (thickness: 50 nm) on the Al₂O₃ substrates, followed by Au thin films (600 nm) in the same chamber. For the RuO₂ deposition, the sputtering pressure was kept at 1.4 Pa in Ar + O₂ (Ar : O₂ = 9 : 1) atmosphere. In the case of Au films, the pressure was kept at 0.7 Pa in Ar atmosphere.

In order to compare with the Au/RuO₂ system, the Au (600 nm)/Pt (150 nm)/Ti (50 nm) system was prepared. These films were deposited on Al₂O₃ substrates on the same conditions for the Au deposition.

2.2 Characterization

The adhesive properties of the deposited films were evaluated by a pull test. Before the test, samples were heated at 200, 400, 500, 600, 700 and 800°C for 1 h in a furnace. Then Au surface was plated with Ni to prevent the reaction between Au thin films and 60 mass% Pb–40 mass% Sn solders, which were used for a Ni pin to be bonded to the Ni surface. The Ni pin was pulled until the Au thin films were removed from the Al₂O₃ substrates and the force to remove the Au thin films from the substrates was measured.

The as-deposited and heat-treated specimens were examined by X-ray diffraction. The heat treatment was done at 700°C for 1 h by a furnace.

To test the thermal stability of the Au/RuO₂ and the Au/Pt/Ti systems, the changes in the sheet resistance of the both systems were measured with a four-point probe. The heat treatment was done by the same way as the pull test.

3. Results and discussion

Figure 1 shows the changes in the pull strength of the Au/RuO₂ and the Au/Pt/Ti systems with temperature up to 800°C. Before heating, the pull strength of the Au/Pt/Ti system is greater than that of the Au/RuO₂ system. The Ti thin film between the Pt thin film and the substrate is found to improve the adhesive properties at room temperature. However, when the temperature goes up to 500°C, the pull strength of the Au/Pt/Ti system is sharply reduced to one-half of its initial value. On the other hand, the pull strength of the Au/RuO₂ system remains constant up to 800°C.

The Au/Pt/Ti system shows that the Ti thin film holds the role of the adhesion layer up to 300°C. In the case of the Au/RuO₂ system, the RuO₂ thin film can maintain the property of the adhesion layer up to 800°C.

Figure 2 shows the X-ray diffraction patterns of the samples. Within Au phases, only the Au(111) peak is observed in the both systems. With increasing the temperature, although the peak position does not change, the peak intensity of the Au(111) peak for the Au/RuO₂ system becomes stronger than that of the as-deposited state. It suggests that the RuO₂ thin films can cause highly oriented Au thin films in the Au(111) plane during the heating. In the case of Au thin films, surface energy is the lowest at the Au (111)

Fig. 1. Changes in the pull strength of the Au/RuO₂ (○) and the Au/Pt/Ti (●) systems with temperature.

Fig. 2. X-ray diffraction patterns of the Au(111) peak for the Au/RuO₂ (a) and the Au/Pt/Ti (b) systems. (-----: as prepared —: heat-treated at 700°C for 1 h).

surface.³⁾ It is supposed that the interfacial free energy between the Au and the RuO₂ thin films is decreased by the heat treatment. This may be one of the reason why the pull strength of the Au/RuO₂ system is high and stable up to 800°C for 1 h.

The peak intensity of the Au(111) peak for the Au/Pt/Ti system becomes weak and the peak shifts to higher 2θ at the same temperature. According to Tisone and Drobek,¹⁾ rapid diffusion of Ti occurred along the grain boundaries of the Au and the Pt thin films and the second phases were formed above 500°C. These phases seem to destroy the highly oriented Au thin films in the Au(111) plane and decrease the adhesive properties to the Al₂O₃ substrates.

Figure 3 shows the effect of the heating time on Au(111) peak intensity for both systems. At 700°C, a heating time of 0.5–1 h is enough to obtain the highly oriented Au thin films of the Au/RuO₂ system.

Figure 4 shows the changes in the sheet resistance of the Au/RuO₂ and the Au/Pt/Ti systems heated up to 800°C for 1 h. The sheet resistance of the Au/RuO₂ system remains constant up to 700°C and increases thereafter. This result shows that interdiffusion at the interface does not occur up to 700°C, but at 800°C, elements such as RuO₂ or compositions of the substrates have diffused into the Au thin films.

On the other hand, the changes in the sheet resistance of the Au/Pt/Ti system starts at 500°C, then it increases with

Fig. 3. Au(111) peak intensity for the Au/RuO₂ (○) and the Au/Pt/Ti (●) systems heated at 700°C as determined by X-ray diffraction.

Fig. 4. Changes in the sheet resistance of the Au/RuO₂ (○) and the Au/Pt/Ti (●) systems with temperature.

temperature. The Pt thin films can not operate as the diffusion barriers above 500°C.

Judging from the results of these experiments, the RuO₂ thin films can be said to serve the heat-resisting adhesion layers for the Au thin films.

4. Conclusions

Changes in the pull strength and the sheet resistance of the Au/RuO₂ system with temperature up to 800°C have been measured. From the experiments, the following have found.

(1) The RuO₂ thin films can enhance the adhesive properties between the Au thin films and the Al₂O₃ substrates. The adhesive properties are not influenced by heating and stable up to 800°C for 1 h. The RuO₂ thin films can operate as the adhesion layers at higher temperatures than the Ti thin films.

(2) The sheet resistance of the Au/RuO₂ system remains constant up to 700°C for 1 h. It is no need to insert the diffusion barriers between the Au thin films and the RuO₂ thin films.

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