Residual stress of Cu/TiN films deposited by ion plating and rf sputtering

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Abstract: The residual stresses and crystallographic structures of Cu/TiN multi-layer films deposited on glass substrates are investigated. The TiN layers are first deposited by arc ion plating onto the substrate, followed by Cu films deposited by plasma coating or RF sputtering. The crystallographic structure and residual stresses in the multi-layer films deposited as a function of the thickness of the TiN layer are then investigated by X-ray diffraction.

The TiN layers deposited by arc ion plating on glass substrate are found to exhibit strong {111} orientation. The Cu layers deposited by both plasma coating and RF sputtering on top of the TiN layers are also found to exhibit strong {111} orientation. The two-exposure method is used to evaluate residual stresses in the Cu and TiN layers by measuring lattice strains in two directions determined from the crystal orientation. In the case of RF sputtering, the Cu and TiN layers in the Cu/TiN film are found to exhibit tensile residual stresses of 450~630 MPa and 230 MPa, respectively. In case of plasma coating, however, the Cu and TiN layers in the Cu/TiN films exhibit tensile residual stresses of 180~240 MPa and 300 MPa, respectively

Key word: Multi layer film, Cu film, TiN film, X-ray diffraction, Crystal structure, Residual stress

1. INTRODUCTION

In recent years, the technology for manufacturing highly integrated circuits has improving remarkably. With this improvement, wiring material has changed from Aluminum to Copper, which is characterized by low electric resistance and high resistance to metal migration. However, because Copper diffuses through the silicon wafer substrate, a layer of TiN or TaN is generally deposited between the Copper and silicon wafer to act as a diffusion barrier. During thin film growth, residual stresses arise from the deposition process, with the residual stresses in thin film coatings depending on the mechanical properties of the materials involved [1-4].

The present study investigates the residual stresses and crystallographic structures of Cu/TiN films deposited onto glass substrates. TiN film is first deposited onto the substrate as an undercoating layer by arc ion plating (AIP), followed by Cu films deposited by plasma coating (PC) and RF sputtering. The crystallographic structure and residual stress of the deposited two-layered film were investigated by X-ray diffraction as a function of the thickness of the TiN layer.

2. EXPERIMENTAL DETAILS

2.1. Deposition of TiN Films

Substrates were glass (Borosilicate glass: Corning 7059) plates of dimensions 50mm × 50mm × 1mm. An AIP system (Kobe Steel Co.) was used to prepare the TiN films on these substrates. Figure 1 shows a schematic of the system. After degassing, nitrogen gas was injected into the chamber at a pressure of 1 Pa. Arc

current of 60A and bias voltage of 0V were used. Films were grown to a thickness of approximately 1.0μm. The substrate surface was located perpendicularly to the Ti target.

Prior to deposition, substrate surfaces were treated by ion cleaning (Ti) to remove impurities. Ion cleaning was then performed twice at an arc current of 60A, a bias voltage of -600V, and a processing time of 2 min. Specimens reached a temperature of about 470K during cleaning.

2.2. Deposition of Cu Films

Cu films were deposited by plasma coating (PC) and RF Sputtering. Figure 2 shows a schematic of the PC

Fig. 1. Schematic illustration of the apparatus for Arc Ion Plating.

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Fig. 2. Schematic illustration of the apparatus for plasma coating.

Fig. 3. Schematic illustration of the apparatus for rf sputtering.

system. A Cu target evaporates in the presence of an Ar plasma produced by a plasma gun. The evaporated Cu metal ionizes in the Ar plasma, is attracted by the bias voltage to the substrate surface and is deposited to form a Cu film. An electric power of 4.4 kW was applied to the plasma, depositing pressure in the chamber was 6.3×10^{-2} Pa, film thickness was approximately 2.0 μm . In the PC, ion cleaning thinks of a thermal influence over lower layer and isn't doing it.

Figure 3 shows a schematic of the RF sputtering system. After degassing, argon gas was injected into the chamber at a pressure of 0.26Pa. Sputtering power and film thickness were 200W and 1.0 μm , respectively. Table 1 shows parameters used to deposit the Cu and TiN layers.

2.3. X-ray Stress Measuring Method

As detailed later, the Cu and TiN films exhibit high {111} orientation. As a result, diffraction peaks only appear at a fixed set of ψ angles depending on the crystal structure and crystal orientation. The $\sin^2\psi$

Table 1. Deposition parameters of the Cu and TiN layer.

Film	Cu		TiN
Depositing method	RF Sputter	PC	AIP
Substrate	Glass		
Arc current(A)	—	—	60
Bias voltage(V)	—	—	0
Sputter power(kW)	0.2	—	—
Discharge power(kW)	—	4.4	—
Depositing pressure (Pa)	0.26	6.3×10^{-3}	1
Film thickness (μm)	1.0	2.0	0, 0.26 0.52, 1.00

Table 2. X-ray diffraction measurement condition.

Material	TiN	Cu
Characteristic X-ray	CuK α	
Tube voltage	35 kV	
Tube current	15 mA	
Filter	Nickel	
Diffraction for stress measurement (2θ)	TiN422 (125.8°)	Cu222 (95.2°)
X-ray optics	Para-focusing	
Irradiated area	$2 \times 6 \text{ mm}^2$	$2 \times 8 \text{ mm}^2$

method, therefore, could not be used to measure the residual stresses in the films, which were instead measured using the two-exposure method in which lattice strain is measured in directions determined by the crystal orientation of the film [5,6]. When the {111} plane of the cubic lattice is parallel with the surface, Cu222 diffraction peaks were found at $\psi = 0^\circ$ and 70.5° and TiN422 diffraction peaks were found at $\psi_1 = 19.5^\circ$ and $\psi_2 = 61.9^\circ$ [7]. In both cases, ψ is the angle between the normal of the specimen surface and the normal of the diffraction plane. Assuming isotropic stress in the plane, the following equation can be used to evaluate the residual stress,

$$\sigma = \frac{1}{S_{44}/2} \cdot \frac{\epsilon_{\psi_1} - \epsilon_{\psi_2}}{\sin^2\psi_1 - \sin^2\psi_2} \quad (1)$$

where S_{44} is the elastic compliance of Copper and TiN single crystal, and the values are $13.26 \times 10^{-3}/\text{GPa}$ [8] and $5.95 \times 10^{-3}/\text{GPa}$ [9], ϵ_{ψ_1} and ϵ_{ψ_2} are lattice strains at the angle of ψ_1 and ψ_2 , respectively. Table 2 shows the X-ray measurement conditions.

Fig. 4. Relationship between the thickness of the TiN layer and diffraction patterns of TiN films on glass substrate.

Fig. 5. Relationship between the thickness of the TiN layer and diffraction patterns of Cu/TiN films on glass substrate, where the Cu films deposited by plasma coating.

3. RESULTS AND DISCUSSION

Figure 4 shows the relationship between film thickness of TiN film and X-ray diffraction pattern (XRD) of TiN films deposited by the AIP on the glass substrate. Because of a highly oriented {111} structure of the TiN film, only two intensity peaks of 111 and 222 diffractions of TiN were detected in θ - 2θ scanning for the TiN film. The results revealed that the peak intensities of TiN111 and 222 diffractions become highly as the increase of film thickness. Therefore, the TiN films have {111} oriented crystall, i.e., the {111} crystals axis of the TiN crystals coincides with the normal of the TiN film surface.

Figure 5 shows the relationship between the thickness of the TiN layer and XRD patterns of the Cu/TiN multi-layer films. Cu films were deposited by PC on top of TiN films of various thickness, with the thickness of all of the Cu films being about $2.0\mu\text{m}$. For the pure Cu film, a large intensity peaks of Cu111 diffraction at $2\theta = 43.3$ were observed. Also, the small intensity peaks of

Fig. 6. Relationship between the thickness of the TiN layer and diffraction patterns of Cu/TiN films on glass substrate, where the Cu films deposited by RF sputtering.

Cu200 and 222 diffraction at $2\theta = 50.5$ and 95.2° were observed. This indicates that the Cu film is mainly composed of {111} oriented crystall, but small parts of the crystal are randomly oriented. For the Cu films deposited on TiN layers, the intensity of Cu(111) and (222) diffraction peaks increase with increasing TiN layer thickness, indicating that the growth of the Cu layer is influenced by the morphology of the TiN layer.

The Cu/TiN films in which the Cu film was deposited by RF sputtering were also examined. The thickness of these Cu film was about $1.0\mu\text{m}$. Figure 6 shows the relationship between film thickness of TiN layer and XRD pattern from Cu/TiN film. For the pure Cu film, a large intensity peaks of Cu111 diffraction at $2\theta = 43.3$ and a small intensity peaks of Cu200 and 222 diffraction at $2\theta = 50.5$ and 95.2° were observed. For the Cu films deposited on TiN layers, the peak intensity of Cu 111 and 222 diffraction increases with increasing the thickness of TiN layer. Thus, the crystallographic properties of the Cu film deposited by RF sputtering exhibited similar tendencies to those deposited by PC.

Figure 7 shows the relationship between the thickness of the TiN layer and residual stresses in the Cu and the TiN layers in the Cu/TiN films. Residual stresses of the Cu and TiN films were measured five times, with the TiN layer only able to be measured for film thicknesses of over $1.0\mu\text{m}$. In the films deposited using RF sputtering, the Cu and TiN layers in the Cu/TiN film exhibited tensile residual stresses of $450 \sim 630\text{MPa}$ and 230MPa , respectively. However, in the films deposited by PC, the Cu and TiN layers exhibited tensile residual stresses of $180 \sim 240\text{MPa}$ and 300MPa , respectively. These stresses in both Cu layer increased with increasing the film thickness of the undercoated TiN layer.

Residual stresses in multi-layer systems are generally caused by both intrinsic stress and thermal stress. Thermal stress can be calculated from differences in the thermal contraction of the upper and lower layers, and is given by

Fig. 7. Relationship between the thickness of the TiN layer and residual stresses in the Cu and TiN layers of Cu/TiN films on glass substrate.

$$\sigma_{th} = \frac{E_{upper}}{1 - \nu_{upper}} (\alpha_{upper} - \alpha_{lower}) \Delta T \quad (2)$$

where E_{upper} and ν_{upper} are Young's modulus and Poisson's ratio of the upper layer. ΔT is the temperature drop from the depositing temperature to the room temperature, and α_{upper} and α_{lower} are the thermal expansion coefficients of the upper and lower layers. The value of α_{Cu} , α_{TiN} and α_{glass} is $16.8 \times 10^{-6} K^{-1}$ [6], $9.35 \times 10^{-6} K^{-1}$ [10] and $4.6 \times 10^{-6} K^{-1}$ [11], respectively.

The thermal stresses suggested by equation (2) are tensile in both the Cu layer deposited on TiN and in the TiN layer deposited on glass. However, in this study, depositing temperature could not be measured. Therefore, the details remain unclear, and further research is necessary.

4. CONCLUSION

The crystalline structure and residual stresses of Cu/TiN films deposited by arc ion plating, plasma coating and RF sputtering were investigated. TiN layers deposited by AIP on the glass substrates were found to exhibit strong {111} orientation, as did both the Cu layers deposited by PC and by RF sputtering on top of the TiN layer.

In the case of RF sputtering, the Cu and TiN layers in Cu/TiN film exhibited tensile residual stresses of 450 ~ 630 MPa and 230 MPa, respectively, whereas in the films deposited by PC, the Cu and TiN layers exhibited tensile residual stresses of 180 ~ 240 MPa and 300 MPa, respectively.

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