PROPERTIES OF PIB-CU FILMS ACCELERATION VOLTAGE AND IONIZATION POTENTIAL

**K. H. Kim, H. G. Jang, S. Han, S. C. Choi, D. J. Choi*,
H. J. Jung, and S. K. Koh**

Div. of Ceramics, Korea Institute of Science and Technology, P. O. Box 131, Cheongryang, Seoul, Korea.
*Dept. of Ceramic engineering, Yonsei university, Seoul, Korea

ABSTRACT

Cu films for future ULSI metallization were prepared by partially ionized beam (PIB) deposition and characterized in terms of preferred orientation, grain size, roughness and resistivity. PIB-Cu films were prepared on Si (100) at pressure of $8 \times 10^{-7} \sim 1 \times 10^{-6}$ Torr. Effects of acceleration voltage and ionization potential on the properties of PIB-Cu films have been investigated. As the acceleration voltage increased at constant ionization potential of 400 V, the degree of preferred orientation and surface smoothness of the Cu film increased. At the ionization potential of 450 V, the degree of preferred orientation at the acceleration voltage higher than 2 kV decreased and surface roughness increased with acceleration voltage. Grain size of Cu films increased to 1100 Å initially up to applied acceleration voltage of 1 kV, above which a little increase occurred with the acceleration voltage. There was no indication of impurities such as C, O in all sample. Resistivity of Cu film had the same trends as the surface roughness with acceleration voltage and ionization potential. The increase of electrical resistivity of PIB-Cu films was explained in terms of grain size and surface roughness

INTRODUCTION

Al and its alloys are susceptible to electromigration and stressmigration failures in future shrinking dimensions. Among metals to replace Al and its alloys, Cu is the most attractive one because Cu has lower bulk resistivity (1.7 $\mu\Omega$ cm) than that of Al and its alloys (2.74-3.5 $\mu\Omega$ cm). In addition, Cu has a high resistance to electromigration compared with Al and its alloys, since it has a high activation energy against electromigration which is twice as large as that of Al. For metallization in the future ultra large scale integration (ULSI), downward scaling of the feature size is essential. Because of reduced metallization line width and elongated line length, electrical resistivity of Al and Al alloys used in the current metallization have limitations for application in the ULSI[1]. Alternative to Al, Cu appears to be promising because of its low electrical resistivity as well as high electromigration resistance. In order to apply Cu as an interconnection material, it is neces-

sary to fabricate Cu films with near bulk resistivity. In a bulk metal, the resistivity is known to occur due to collisions of conduction electrons with phonons, impurities, and defects. Since dimensions of a thin film are small relative to bulk, surface scattering and grain boundary scattering contribute largely to increase of resistivity[2,3]. Many articles have been written on theories of resistivity in thin films, in which resistivity was affected by such properties as grain size and surface morphology[2-5]. Even though properties of thin films are influenced by deposition technique and deposition conditions, effects of deposition parameters on resistivity and other properties are not well delineated to date.

In this study, Cu films were prepared by a partially ionized beam deposition(PIBD) technique[6]. The PIBD technique in preparation of metal films is capable of depositing high quality films with good properties such as adhesion, thermal stability, and crystallinity[7-10]. The primary purpose of this paper is to advance understanding of the relationship between electrical resistivity and the properties of Cu films grown by PIB deposition, such as grain size and surface roughness which were characterized by Auger electron spectroscopy (AES), scanning electron microscopy (SEM) and atomic force microscopy (AFM).

EXPERIMENTAL

The PIB source for deposition of Cu thin films consisted of three main parts : metal evaporation, ionization, and acceleration. Metal evaporation part employed a graphite crucible with a cylindrical nozzle of which dimension is $1 \times \varphi 1 \, mm^2$ and a filament for electron ejection. The crucible was heated by bombardment of electron emitted from the filament. Vaporized metal particles were ejected through the nozzle. The crucible was preheated at 2000℃ for 2 hours to eliminate carbon contamination during evaporation of Cu. Crucible temperature during the deposition was set at around 1700℃ measured by optical pyrometer (MINOLTA SPOT THERMOMETER IR-630).

The vaporized metal particles were partially ionized by electron impact collision in the ionization region. Ionized particles were accelerated by acceleration potential applied between the crucible and the accelerating electrode. In ionization and acceleration processes, ionization potential controlled ionization efficiency of particles and average energies of depositing particles depended on the acceleration voltage as well as the ionization potential. The ionization potential and the acceleration voltage were taken as experimental parameters in this experiments. The crystallinity, microstructure and surface roughness of the film were varied with deposition parameters. Deposition conditions are summarized in Table I.

Table I. Deposition conditions for Cu films

Parameter	Condition
Working pressure	$8 \times 10^{-7} \sim 1 \times 10^{-6}$ Torr
Substrate temperature	Room temperature
Crucible temperature	about 1700℃
Ionization potential and current	400V(300mA) and 450V(350mA)
Acceleration voltage	$0 \sim 3$ kV

The source material for evaporation was Cu (99.999 %. Rare Metallic Co., LTD). Si (100) wafer was used as the substrate. The Si (100) substrate was placed at 40 ± 0.3cm above the top of the PIB source. The substrate was cleaned by acetone and ethanol, and dried by pure N_2 gas. The deposition was performed at pressure of $8 \times 10^{-7} \sim 1 \times 10^{-6}$ Torr. Temperature of the substrate was kept at room temperature.

The deposition rate and the thickness of Cu films were measured, respectively, with crystal quarts monitor (MAXTEK Inco., TM-103R) which were calibrated by RBS and profilometer (α-step 200, Tencor Co.). The deposition rates varied from 18Å/min to 20 Å/min and thickness of all Cu films was 1800Å. AES was used to determine impurity concentration in the Cu films. The crystal structure of the Cu films was investigated by X-ray diffraction (Philips, PW7602) analysis. Surface morphology and surface roughness of the Cu films were investigated by SEM and AFM, respectively. The grain size of the Cu films was determined by SEM and AFM. The resistivity of Cu films was measured by a four point probe method.

RESULTS AND DISCUSSIONS

All PIB-grown Cu films have highly $\langle 111 \rangle$ preferred orientation and the degree of preferred orientation depends on V_a and V_i. Figure 1 shows the relative intensity ratio, $I(111)/I(200)$, of the Cu films obtained by XRD as a function of V_a for Vi = 400 V and 450 V. The relative intensity ratio of the Cu films deposited at $V_i = 400$
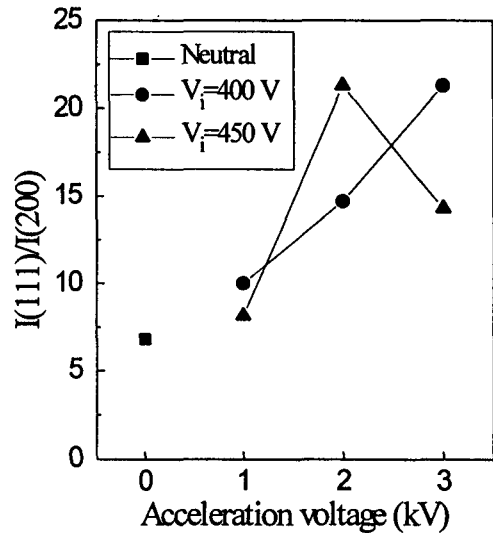

Fig. 1. The x-ray relative intensity ratio, I(111)/I (200) of Cu films with acceleration voltge

V increases from 6.8 to 21 with increase of the V_a from $V_a = 1$ kV to $V_a = 3$ kV. However, $I(111)/I(200)$ of Cu films deposited at Vi = 450 V increases up to 21 at Va = 2 kV and then decreases to 14 at Va = 3 kV. As reported by Koh. et al[6], the degree of preferred orientation was affected mainly by the average energy per depositing atom rather than by only bias voltage. The average energy is a function of the acceleration voltage and ionization potential. The average energy of about 60 eV/atom for highly $\langle 111 \rangle$ oriented Cu films might be the optimum energy.

No impurities were detected within the limitations of AES (not shown here). All the Cu films deposited at various conditions showed also the same results. Impurities such as C, O in thin metal films give steep rise in the resistivity[11]. Since all kinds of impurities were hardly found in PIB-grown Cu films, the resistivity of the films arising from the impurities is negligible. Impurities such as C, O were observed in MOCVD[12] and Ar in spu-

ttering system or ion assisted deposition[13-14].

The Cu films appears to have dense structures. There are no such a voids and crack as can be observed. Changes of average grain size as a function of V_a and V_i are shown in Fig. 2.

The average grain size of Cu film grown at $V_a=0$ kV is measured to be around 620 Å. The average grain size of the Cu films deposited with $V_a=1$ kV to 3 kV at $V_i=400$ V ranges from 1093 Å to 1919 Å. It can be seen in Fig. 2 that the grain grow with V_a at both $V_i=400$ V and $V_i=450$ V. It increases from 620 Å at $V_a=0$ kV to 1100 Å at $V_i=400$ V and 1160 Å at $V_i=450$ V. The average grain size shows the maximum sizes of 1919 Å at $V_i=400$ V and 1440 Å at $V_i=450$ V with V_a $=2$ kV. It then actually decreases to 1500 Å at $V_i=400$ V and 1330 Å at $V_i=450$ V with $V_a=3$ kV. The grain growth behavior might be due to a combination of the adatom migra tion enhanced with increasing the Va[8] and

the increase of nucleation sites number, with ion beam current density. However, when the number of nucleation sites exceeds a certain value with increasing Va, grain size of film would then decrease[15-16]. There might be an optimum condition to get large grain size between adatoms migration and concentration of nucleation sites.

Figure 3 shows surface roughness of Cu films with various acceleration voltage at V_i $=400$ V and $V_i=450$ V. The root mean square (R_{ms}) surface roughness, σ, of PIB-Cu films decreases with increasing V_a at $V_i=400$ V, but the σ's increases with acceleration vol-tage at $V_i=450$ V. These changes of surface roughness might be due to resputtering of Cu films by ion beam as well as grain structure of PIB-Cu films[15].

Resistivity of Cu films at $V_i=400$ V and V_i $=450$ V is plotted in Fig. 4 as a function of acceleration voltage. For the Cu films at $V_i=$ 400 V, the resistivities of Cu films were approaching to bulk value with the acceleration
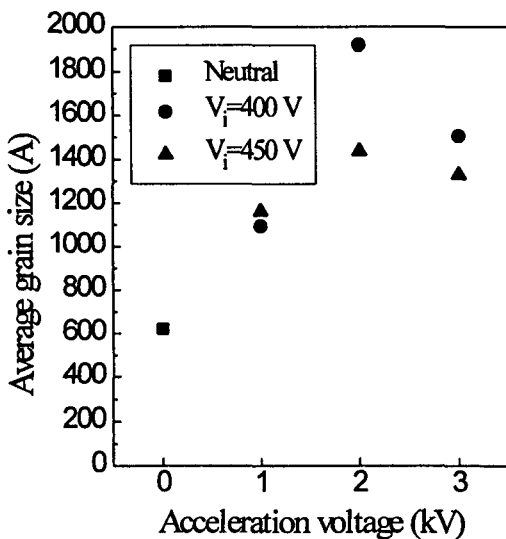
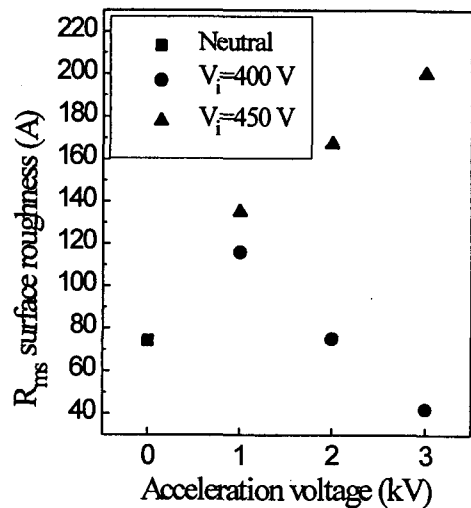Fig. 2 The average grain size of Cu films at ionization potential of 400 and 450 V with acceleration voltage.

Fig. 3 The root mean square(R_{ms}) surface roughness of Cu films at ionization potential of 400 and 450V with acceleration voltage.

voltage. The minimum value of 1800 thick Cu films was found to be 1.94 $\mu\Omega$·cm at $V_a=3$ kV, very close to the bulk resistivity ($1.7\mu\Omega$·cm). Results at $V_i=450$ V represented the opposite to those at $V_i=400$ V, where the resistivities of Cu films at $V_i=450$ V increase with increasing V_a.

In thin metal films, resistivity of film is generally higher than bulk resistivity. These behaviors are caused by scattering of conducting electron from scattering from surfaces[2] and scattering from grain boundary[3]. According to Matthiessens rule, total resistivity of metal thin film is represented as ;

$$\rho_T = \rho_B + \rho_I + \rho_S + \rho_{G\cdot B} \tag{1}$$

where, $\rho_B\rho_I$, ρ_S, $\rho_{G.B}$., are the individual bulk, impurity, surface scattering and grain boundary resistivities, respectively.

From eq. (1), it is considered that a resistivity higher than the bulk value is mainly due to three causes, ρ_I, ρ_S, $\rho_{G.B}$. All PIB-Cu films deposited at various V_i and V_a do not include impurities within AES detection limitation. Assuming that Cu films have the same impurity concentration as Cu source material (5N), it is considered that scattering from impurity may give little effect to change of resistivity in Cu films by PIB. In order to investigate the contribution from $\rho_{G.B}$, the ratio of grain boundary to background resistivity was calculated by inserting average grain size of Cu films by PIB into eq. (2) formulated by Mayadas and Shatzkes[3].

$$\frac{\rho_0}{\rho_{G.B}} = 3\left[\frac{1}{3} - \frac{1}{2}\alpha + \alpha^2 - \alpha^3 \ln\left(1 + \frac{1}{\alpha}\right)\right] \tag{2}$$

where, $\quad \alpha = \frac{\lambda_0}{r} \times \frac{R}{(1-R)}$

r : average grain size
R : grain boundary reflection coefficient
λ_0 : electron mean free path in the metal
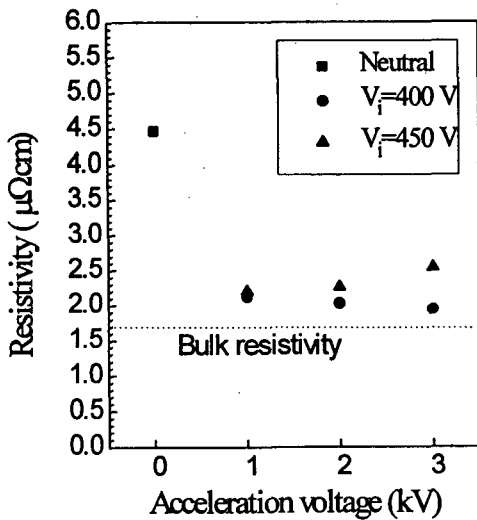   (382.8 Å for Cu)

Fig. 4 The resistivity of Cu films at ionization potential of 400 and 450V with acceleration voltage
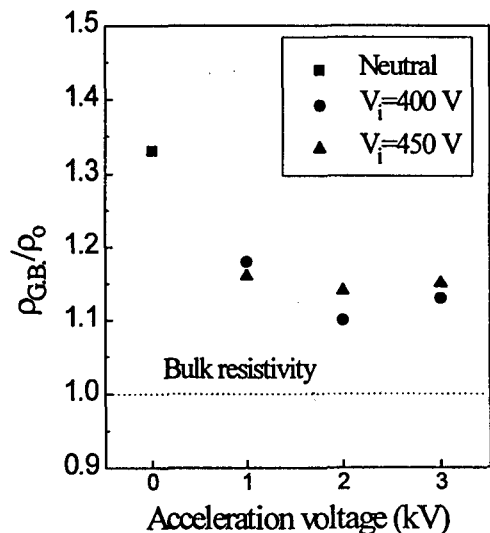

Fig. 5. The grain boundary enhanced resistivity to the bulk resistivity at ionization potential of 400 and 450 V with acceleration voltage.

Even though R is dependent on grain boundary type, the intergrain geometry and radius of curvature[18], we have taken R as 0.24 for simplicity. These results are shown in Fig. 5.

It is shown from Fig. 5 that grain size as large as 620 Å give rise to increment of 33 % in resistivity and grain size larger than about 1000 Å at $V_i = 400$ V and 450 V with V_a contributes to resistivity with amount of 10 to 18 %. Fuchs[2] proposed a model that as thickness of thin metal film decrease, surface scattering of conducting electron increase resistivity. However, Ziman[4] and Soffer[5] reported that surface roughness was the cause of diffuse scattering and as the surface roughness increase, diffuse scattering at surface increase resistivity. Comparison of the surface roughness and the resistivity of Cu films by PIB may reflect the fact that surface roughness cause diffuse scattering to increase resistivity. Definitely to show relation surface roughness with resistivity, the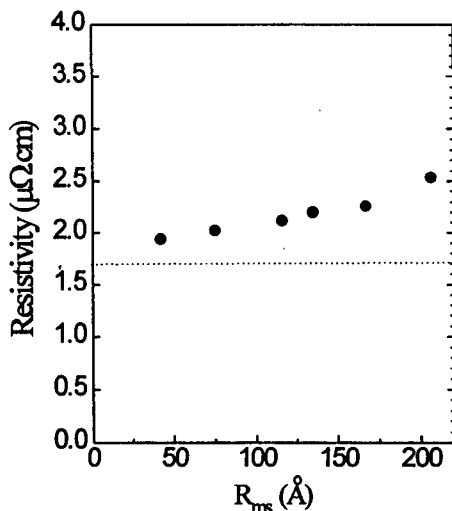 resistivity is shown as a function of surface roughness in Fig. 6 where data at neutral is excluded because it is dominantly affected by grain boundary scattering. Even if experimental contribution from ρ_s is higher than the value approached by theoretical calculation, these results could show effects of the surface roughness on resistivity. Consequently, surface roughness among sources causing scattering such as impurity, grain boundary and surface might give dominant effects to resistivity of Cu films deposited by PIB.

CONCLUSIONS

Cu films were prepared on Si(100) by partially ionized beam(PIB) deposition at pressure of $8 \times 10^{-7} \sim 1 \times 10^{-6}$ Torr. As the acceleration voltage increased at ionization potential of 400 V, the degree of preferred orientation and smoothness of Cu films increased. At ionization potential of 450 V, degree of orientation at acceleration voltage above 2 kV decreases and the surface roughness increases with the acceleration voltage. A large increase in grain size of Cu films occurs initially up to applying acceleration voltage of 1 kV, above which little increase occurs with the acceleration voltage. The resistivity of Cu film had the same tendency as surface roughness with the acceleration voltage and ionization potential. It is rather surface roughness than grain size or impurity level to substantially affect changes of resistivity in Cu films deposited by PIB.

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Fig 6. The resistivity of Cu films with the surface roughness

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