Original Received December 27, 2005 Accepted for Publication March 9, 2006 © 2006 Soc. Mater. Eng. Resour. Japan

Inference of Current density on Microstructure of Electroplated Cu Thin Film

T. MASAI, K. TAKASUGI, K. -K. CHOI and S. SATO

Advanced Process Technology Center, TDK Corporation, 2-15-7, Higashi-Ohwada, Ichikawa-shi, Chiba 272-8558 Japan *E-mail : masai@mb1.tdk.co.jp*

In this paper, we report micro-structural characteristics of electroplated Cu thin films with the variation of the current density applied during the electroplating process. We evaluated the surface roughness, the crystalline texture, the resistivity, and the grain size of the thin film in a wide range of current density $(50-1000 \text{A/m}^2)$. The surface roughness and the resistivity were increased according to the increment of the current density. The $\langle 111 \rangle$ textured structure was also pronounced as the current density was increased. The scanning ion microscope (SIM) image of the cross-sectional samples revealed that grain size of the film prepared in a high current density was much smaller $(0.05 \sim 0.5 \mu \text{ m})$ than that of the thin film in a low current density $(1 \sim 2\mu \text{ m})$. These results are different from the film prepared by a sputtering process, where a smooth surface was observed in a small grain-sized ($\langle 0.1 \mu \text{ m} \rangle$ film.

Key Words : Cu electroplating, thin film, grain size, current density

1. Introduction

For the purpose of reducing the loss of current, Cu electroplated thin film is actively used in LSI as a interconnect metal instead of Al [1]. In the damascene process, the thickness of Cu electroplated thin film is under 100 nm order. Therefore, the demanded reliability level of interconnect becomes severe year by year.

Cu electroplated thin film is used on not only LSI but also MEMS (Micro Electro Mechanical Systems) devices. For example, electroplated thin films are used as signal lines or beams of the MEMS devices [2]. MEMS device has the possibility of integration of passive devices with active device such as RF or sensor modules. In order to draw out the device performance, it is important to understand the relation between the process parameter and the characteristics of electroplated materials used in the device.

In this paper, we investigate the relation between the applied current density during electroplating process and the characteristic of electroplated thin film. The surface roughness, the crystalline texture and the grain size are observed for electroplated Cu films of different applied current density. Microstructure of sputter deposited Cu films is also observed for comparison.

Experimental procedure

The seed layer for electroplating, which thickness is 50 nm of Ti layer and 250 nm of Cu layer, was deposited on the oxidized Si wafer by the magnetron sputtering apparatus. 5μ m thickness of the Cu thin films were electroplated on the seed layer in the sulfuric acid bath at room temperature. The current density was 78, 156, 311, 467, 623, and 934 A/m². Other parameters such as the motor speed for the rolling the wafer and the flow rate of the circulation were fixed. In order to deposit 5μ m thickness of Cu thin film, the deposition time is determined according to the deposition rate of each current density.

The crystalline texture of electroplated Cu thin film was characterized by X-ray diffraction (XRD) measurements. The surface morphology was evaluated by scanning electron microscope (SEM) and atomic force microscope (AFM). Furthermore, the scanning ion microscope (SIM) images were observed for the cross-sectional samples prepared by focused ion beam (FIB) apparatus. The resistivity was measured by four-point probing system.

3. Results and discussion

3.1 Surface morphology

The surface morphology as a function of applied current density for electroplated Cu thin film observed by SEM and AFM is shown in Figure 1 and Figure 2, respectively. An AFM image of sputter deposited Cu film is shown in Figure 2 (g) for comparison. The average surface roughness (Ra) observed by AFM is shown in Figure 2.

In the range of low current density (under 311 A/m²), the surface is smooth without large protrusions. However, protrusions appear in the range of middle current density (around 467 A/m²). And then, large protrusions $(0.5 \mu \text{m} \text{ to } 1 \mu \text{m} \text{ of diameter})$ are observed at the current density over 623 A/m².

The current density dependence in the average surface roughness observed by AFM is plotted in Figure 3. The solid line indicates the surface roughness of sputter deposited thin film (7.3

nm). The surface roughness is under 20 nm in the range of low current density ($<311 \text{ A/m}^2$). An abrupt increase of surface roughness (Ra>100 nm) is observed when the current density exceeds 623 A/m². It is hard to explain this phenomenon clearly, although there is the influence of additives in the sulfuric acid bath on the roughness. It is generally said that the leveler adheres to only protrusions of surface, and reduce the speed of deposition [3]. At high current density, it seems that the effect of leveler is small.

We examined the relation between the resisitivity of thin film and the current density as shown in Figure 4. The solid line and the dotted line shows the resistivity of bulk Cu $(1.76 \,\mu \,\Omega \,\mathrm{cm})$ and sputtered thin film $(2.37 \,\mu \,\Omega \,\mathrm{cm})$. In the range of low and middle current density (<467 A/m²), the resistivity is stable at around $1.8 \,\mu \,\Omega \,\mathrm{cm}$. The resistivity is abruptly increased when the applied current density is over 623 A/m². This tendency is similar to the surface roughness as shown in Figure 3, indicating that the surface roughness corresponds to the resistivity.

3.2 Grain texture

The SIM images of cross-sectional samples are shown in Figure 5. Even though precise grain size can not defined by using



Figure 1 The SEM images of the surface morphology as a function of current density (a) 78 A/m², (b) 156 A/m², (c) 311 A/m², (d) 467 A/m², (e) 623 A/m², and (f) 934 A/m²





Figure 3 The current density dependence in the surface roughness of electroplated Cu thin film



Figure 4 The current density dependence in resistivity

98

SIM image because the grains with same orientation appears in the same contrast, clear difference in grain size is observed in this observation.

At 78 A/m² (Figure 5(a)) and 156 A/m² (Figure 5(b)) of current density, the grain size is estimated between 1μ m and 2 μ m. On the other hand, at 934 A/m² of current density (Figure 5(c)), the grain size reduces from 0.05μ m to 0.5μ m.

The deposition mechanism of electroplating thin film has been discussed [3][4]. One of predominant theory [5][6] explains



Figure 5 The cross-sectional SIM image of Cu thin film(a) Electroplated at 78 A/m² of current density (b) Electroplated at 156 A/m² of current density (c) Electroplated at 934 A/m² of current density (d) Sputter deposited film sputtered thin film

that the grain size depends on the relation between the speed of the grain growth and the deposition speed. In the case of high current density, the velocity of the deposition (V_a) is larger than the velocity of the grain growth (V_g) . As the result, the particle will be deposited without the grain growth. On the other hand, if V_a is smaller than V_g , the grain growth is promoted. This mechanism of deposition is common to the growth theory of sputter deposited thin film.

Considering the results of surface roughness, resistivity and grain size (Figures 3, 4, and 5), it seems that Cu films with finer grains prepared at higher current density are coarse and resistive. Even though we mentioned the influence of surface roughness on the resistivity in chapter 3.1, these results also indicate the possibility that the grain boundaries disturb the conduction of electrons.

We also measured the X-ray diffraction (XRD) in order to investigate the texture of Cu thin films. Similar diffraction patterns were observed for all whole range of current density. In Figure 6, the XRD pattern of 156 A/m² of current density is shown as an example. Figure 6 (a) and (b) shows that the $\langle 111 \rangle$ texture was pronounced compared with others. In the fcc (face centered cubic) structure, $\langle 111 \rangle$ texture is pronounced. In order to evaluate the texture structure, Willson's method [3] is used. In this method, the relative intensity of $\langle 111 \rangle$ is calculated using following equation (1).

$$X_{\langle 111\rangle} = \frac{IF_{\langle 111\rangle}}{IFR_{Bulk\langle 111\rangle}}$$
(1)

Where, X $\langle 111\rangle$ is defined as relative intensity of $\langle 111\rangle.$ In addition,

$$IF_{\langle III\rangle} = \frac{I_{\langle III\rangle}}{\sum\limits_{yk} I_{\langle yk\rangle}}$$
(2)



Figure 6 The XRD patterns of 156 A/m² of current density a) Original scale b) Expanded scale $(\times 30)$

$$IFR_{\langle 111\rangle} = \frac{I_{Bulk\langle 111\rangle}}{\sum_{ijk} I_{Bulk\langle ijk\rangle}}$$
(3)

Where I_{ijk} is the intensity obtained by the XRD measurement, and $I_{Bulk \langle ijk \rangle}$ is the intensity obtained by JCPDS data of polycrystalline powder (JPCDS No.; 4-0836).

The current density dependence in relative intensity of texture is shown in Figure 7.

Figure 7 shows that $\langle 111 \rangle$ texture is pronounced over whole range of current density in this experiment. Moreover, the increment of current intensity causes the increment of intensity of $\langle 111 \rangle$ texture.

We compared the characteristics of the electroplated thin film with the characteristics of sputter deposited thin film. The AFM image of sputter deposited Cu film (Figure 2(g)) shows clear grain morphology compared with electroplated films.

The SIM image of sputter deposited Cu film (Figure 5(d)) shows small grains (under $0.1 \,\mu$ m) compared with the electroplated films. The result of XRD patterns show that the percentage of $\langle 111 \rangle$ texture of sputter deposited thin film is lower than that of the electroplated thin film. In contrast, the other texture such as $\langle 311 \rangle$, $\langle 110 \rangle$, and $\langle 100 \rangle$ appeared. The resistivity of sputter deposited thin film is $2.37 \mu \Omega$ cm. This result indicates the possibility that the high resistivity is caused by grain boundary.



Figure 7 Texture of electroplated Cu thin film as a function of current density (Sputter deposited sample (SPT) is also shown comparison.)

In summary, the increment of the current density of electroplating caused high resistivity, fine crystallization, and rough surface. Considering the results of the AFM images and SIM images, the small grains of sputter deposited thin film form the smooth surface roughness. On the other hand, the large grains of the electroplated thin film form the smooth surface roughness. The phenomenon of the surface roughness in electroplated thin film is different from that of sputter deposited thin films.

4. Conclusions

We investigated the relation between the current density and the characteristics of electroplated thin film prepared in the sulfuric acid bath at room temperature. The increment of the current density causes the fine crystallization, the coarse surface roughness, the increment of intensity of $\langle 111 \rangle$ texture, and high resistivity. The average surface roughness in the low current density is 5 times smaller than that in high current density.

We compared microstructure of electroplated thin film with that of sputter deposited thin film. When both grains of sputter deposited and electroplated thin film are small, even though the roughness of electroplated thin film has rough surface, the roughness of sputter deposited thin film has smooth surface.

References

- D. Walther, M. E. Gross, K. Evans-Lutterodt, W.L. Brown, M. Oh, S. Merchant, P. Naresh, "Room temperature Tecrystallization of Electroplated Copper Thin Films: Methods and Mechanisms," Mater. Res. Soc. Proc., Vol.612, (2000).
- [2] P. M. Zavracky, S. Majumder, and McGruer, "Micromechanical Switches Fabricated Using Nickel Surface Micromachining," J. Micro Electromech. Systems, Vol.6, No. 1, pp3-9, (1997).
- [3] T. Watanabe, "Nano-Plating-Microstructure Control Theory of Plated Film and Data Base of Plated Film Microstructure, " Elsevier Ltd., Oxford, (2004)
- [4] Y. Nishihama, "Electrolytic Copper Plating" Journal of the Surf. Finish. of soc. of Jpn., Vol.50, No.2, pp135-139, (1999)
- [5] I. Ohno, S. Haruyama, "Electodeposition of Compound Semiconductor," J Jpn. Inst. Metal, Vol.30, pp735-742, (1991)
- [6] S.Yoshida, "Thin films," baifukan Ltd., (1990)