Improvement of Ta Barrier Film Properties in Cu Interconnection by Using a Non-mass Separated Ion Beam Deposition Method^{*1}

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Ta/Si (100) and Cu/Ta/Si (100) film structures were fabricated by using ion beam deposition with a modified RF sputter-type ion source, in which a strong RF discharge was introduced in order to enhance the plasma density. For Ta/Si structures, Ta films were deposited at various bias voltages. When the substrate bias voltage was not applied, the Ta film showed a columnar structure and had a high resistivity of 2600 nΩm. On the other hand, when the substrate bias voltage of -50–-200 V was applied, the cross-sectional observation did not show columnar structure at all. In this case, film deposition was considered to be sufficient migration energy by the accelerated Ta⁺ ions. In particular, Ta films deposited at a bias voltage of -125 V had a very small resistivity of 360 nΩm. Thermal stability of Cu(100 nm)/Ta(50 nm)/Si films, where Ta plays a role of diffusion barrier, was evaluated after annealing in H₂ atmosphere for 60 min at various temperatures. Non-columnar structure Ta films deposited at substrate bias voltages of -50 V and -125 V were found to be stable up to 600°C, while columnar structure Ta films deposited at zero bias voltage degraded at 300°C. This result indicates that the thermal stability of the Ta films is mainly governed by the film microstructure of the deposited layer.

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1. Introduction

Copper has been gaining wider attention as a new interconnection material of ULSI because of its lower bulk resistivity and higher resistance to electromigration than aluminum and aluminum alloys.^{1,2)} However, we have to overcome disadvantages that copper atoms easily diffuse into silicon through SiO₂ film and create deep trap levels, which result in serious degradation of the device reliability.³⁾ Therefore, an effective diffusion barrier is necessary to suppress copper diffusion into silicon. Tantalum has a suitable property as a barrier material, because tantalum does not react with copper, providing a relatively stable interface between copper and tantalum. Results reported on the failure temperature of tantalum barriers by various fabrication methods ranging from 300–650°C are listed in Table 1.^{4–9)} In the case of the lower failure temperatures, it is considered that fast diffusion of copper atoms through structural defects, such as grain boundaries, results in decreasing the temperature. Therefore, it is expected that eliminating columnar structure by the migration energy of surface atoms with the ion bombardment would enhance the thermal stability.

The IBD method enables us to form high purity and low defect density films, because the kinetic energy of accelerated ions is effectively converted to the migration of atoms on the surface.^{10–14)} In our previous work,¹⁵⁾ we demonstrated that Cu films, deposited by IBD system with a newly developed RF sputter-type ion source, had a very small resistivity and good surface morphology at a bias voltage of -50 V. In the

present work, we investigated the effect of the bias voltages on the resistivity and the microstructure of Ta/Si films and also on the thermal stability of Cu/Ta/Si structures.

2. Experimental Procedures

A non-mass separated IBD system with a newly developed RF sputter-type ion source was already described elsewhere.¹⁶⁾ A base pressure of 10⁻⁵ Pa was obtained in the deposition chamber using a turbo molecular pump. Targets (Cu: 99.9999%, Ta: 99.99%) were electrolytically polished to eliminate the surface contamination and Si (100) substrate was ultrasonically cleaned in acetone and then etched in 5% HF solution. A high purity Ar (99.9995%) gas plasma was initially generated in the ion source by applying a 13.56 MHz RF power to the copper coil. By applying a negative bias voltage of -300 V or -500 V to the copper or tantalum target, respectively, copper- or tantalum-rich plasmas were generated. The distance between the target and the substrate was 35 mm.

In order to fabricate Cu/Ta/Si (100) structures, a Ta film was initially deposited on Si (100) substrate by using a bias voltage ranged from 0 to -200 V at an Ar pressure of 9 Pa for 10 min, and then a Cu (100 nm) film was deposited on the Ta(50 nm)/Si at a voltage of -50 V. In addition, in order to investigate thermal stability of Cu/Ta/Si structures, Cu/Ta(zero bias)/Si and Cu/Ta(biased)/Si structures were annealed for 60 min at temperatures up to 700°C in H₂ atmosphere. Resistivity of deposited films and annealed samples was measured with Van der Pauw method. Phase change of the deposited films was investigated by XRD (Rigaku RINT 2000), and the surface and cross-sectional morphology of the films was observed using FE-SEM (Hitachi S-4100L).

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Table 1 Failure temperatures of Ta diffusion barrier in Cu/Ta/Si structures.

Sample structure (nm)	Fabrication method	Annealing atmosphere	Annealing time (min)	Failure temperature (°C)	Ref.
Cu(200)/Ta(100)/Si	Electron beam evaporation	N ₂ :H ₂ (9:1)	30	300–400	4)
Cu(200)/Ta(180)/Si	Sputtering	Vacuum (10 ^{−5} Pa)	30	550	5)
Cu(100)/Ta(50)/Si	Magnetron sputtering	He	30	500–550	6)
Cu(200)/Ta(100)/Si	sputtering	Vacuum (10 ^{−5} Pa)	60	600	7)
Cu(100)/Ta(30)/Si	Electron beam evaporation	Vacuum (10 ^{−4} Pa)	30	550	8)
	Electron beam evaporation + ion beam assisted deposition	Vacuum (10 ^{−4} Pa)	30	650	
Cu(50)/Ta(Vb)(50)/Si	Optimum bias sputtering	N ₂	30	650	9)
Cu(100)/Ta(unbiased)(50)/Si	Ion beam deposition	H ₂	60	Below 300	This
Cu(100)/Ta(biased)(50)/Si	Ion beam deposition	H ₂	60	600	work

Fig. 1 Resistivity change of Ta films as a function of the bias voltage.

3. Results and Discussion

Ta films were deposited for 10 min on Si (100) substrates at various bias voltages ranged from 0 to −200 V under the following condition: RF power (240 W), target voltage (−500 V) and Ar pressure (9 Pa). Figure 1 shows resistivity change as a function of the bias voltage. A Ta film deposited at zero voltage shows the highest resistivity (2600 nΩm), which is considered as of reported β-Ta films of 1100–3000 nΩm.¹⁷⁾ When the bias voltage was applied to a substrate, the resistivity of the film sharply decreased and at a bias voltage of −125 V, the resistivity had a smallest value of 360 nΩm, which was roughly close to the bulk resistivity of 130 nΩm.¹⁷⁾ It is worth noting that the resistivity of Ta films deposited by IBD system with the high purity ion source has a value roughly equal to the bulk one, which is smaller than the reported value on α-Ta films of 250–500 nΩm.¹⁷⁾ Further increase of the bias voltage up to −200 V, results in gradually increasing the resistivity.

To find out a correlation between the resistivity of Ta films and their microstructure change, we measured X-ray diffraction patterns of the samples deposited at various bias voltages as shown in Fig. 2. In the case of the zero bias voltage, very broad Ta peak was observed. In spite of the presence of a slight peak shift, this corresponds to β-Ta, judging from the measured resistivity value. With increasing the bias voltage,

Fig. 2 X-ray diffraction patterns of Ta films as a function of the bias voltage.

β-Ta (200) peak appeared clearly and became dominant. At a bias voltage of −75 V, α-Ta (110) peak started to appear in addition to the strong β-Ta (200) peak. When the bias voltage was increased to −125 V, β-Ta (200) peak was weakened and the α-Ta (110) peak became the strongest. This result coincides with the fact that electrical resistivity measured on the film deposited at a bias voltage of −125 V was 360 nΩm, which corresponds to the resistivity value reported on α-Ta films of ~500 nΩm.¹⁷⁾ These results make us suggesting that ion bombardment by applying a substrate bias voltage during the deposition process plays an important role in the growth of Ta films. By further increasing in the bias voltage, β-Ta (200) peak intensity again gradually increased and α-Ta (110) peak finally disappeared at a bias voltage of −200 V. This behavior is in a good correspondence with the results of the resistivity measurement as mentioned above.

To confirm the effect of the bias voltage on the surface morphology and microstructure, as well as their correlation with the electrical resistivity, we carried out SEM observation of surface and cross section of the samples deposited at bias voltages of 0, −50 and −125 V as shown in Fig. 3. In Figs. 3(a) and (b), at the zero bias voltage, small grains and cracks were observed clearly on the surface and a columnar structure was found in the cross sectional observation. These results,

Fig. 3 Surface and cross-sectional observations of Ta films at various bias voltages: (a) and (b) 0 V, (c) and (d) -50 V, (e) and (f) -125 V, respectively.

similar to the previous Cu/Si (100) case,⁹⁾ seem to be due to insufficient surface migration of low energy particles, which are considered mainly to be composed of Ta neutral atoms. When the bias voltage of -50 V was applied to the substrate, no cracks were observed, and the surface morphology was remarkably improved as shown in Fig. 3(c). As shown in Fig. 3(d), its cross sectional morphology shows non-columnar structure, which indicates sufficient migration energy by the accelerated Ta^+ ions. The effect of ion bombardment was also investigated by Hirsch *et al.*¹⁸⁾ and proved that when part of the surface was bombarded by argon ions on the germanium films, the deposit showed no sign of flaking or fracture anywhere on the irradiated area. Further increase in the bias voltage to -125 V, which formed dominant α -Ta film, the surface of the film became slightly rougher as shown in Figs. 3(e) and (f). This is explained by a resputtering due to higher energy particle bombardment on the surface during deposition.

In general, sputtered Ta films on silicon substrates prefer to have a metastable β -Ta structure,¹⁷⁾ whereas α -Ta structure can be obtained by served methods, such as applying a bias voltage to the substrate, deposition onto the titanium substrate,¹⁹⁾ annealing treatment,²⁰⁾ and so on. In the present study, we tried to investigate the effect of the substrate bias voltage on the structure of Ta films, and found that it could control the phase of the deposited films, in which ion bombardment played an important role in the film growth.

For Ta/Si structures, it was found that the resistivity of α -phase dominant Ta films deposited at a bias voltage of -125 V had a smallest value (360 n Ω m) and applying a bias voltage of -50 V made the film surface extremely smooth. Thus, the role of Ta film microstructures was studied on the thermal stability of the deposited Cu(100 nm)/Ta(50 nm)/Si structure which are expected to be suitable for a diffusion barrier. Cu films were deposited on Ta/Si substrates at an optimum

Fig. 4 Resistivity as a function of annealing temperature in H_2 for 60 min: (a) Cu(-50 V)/Ta(zero bias)/Si, (b) Cu(-50 V)/Ta(-50 V)/Si, and (c) Cu(-50 V)/Ta(-125 V)/Si structures.

bias voltage of -50 V, which was determined in our previous study.⁹⁾ The optimum applied bias voltage of -50 V have been found to prepare Cu films with non-columnar structure, a very low electrical resistivity (17.9 n Ω m) and smooth surface. Figure 4 shows the resistivity change after annealing at different temperatures for Cu(-50 V)/Ta(zero bias)/Si and Cu(-50 V)/Ta(biased)/Si structures. For the former structure, a sudden sharp increase in the resistivity were observed at about 300°C, suggesting that copper reacted with silicon forms Cu_3Si precipitate at temperatures as low as 300°C.⁵⁾ On the other hand, the latter structures were stable up to 600°C. This increasing in failure temperature must be attributed to the suppression of the copper diffusion through tantalum barrier and indicate the same failure temperature for both α -Ta and β -Ta films means that the thermal stability is not influenced by deposited Ta phases.

There have been reported that formation of a metal silicide is initiated by fast diffusion of silicon through structural defects such as grain boundaries in the metal film.⁸⁾ Thus, the columnar structure in the Ta films deposited at zero bias voltage resulted in the lower failure temperature by the diffusion of Si through Ta grain boundaries. Whereas, Ta films with non-columnar structure deposited at a bias voltage of -50 V or -125 V, improved the thermal stability up to 600°C without reaction of copper with silicon. These results suggest that the stability of the Ta thin films as diffusion barrier layer is mainly governed by the film microstructure of the deposited layer, which can be controlled by the migration energy of surface atoms, caused by the irradiation of Cu neutral atoms or accelerated Cu^+ ions.

4. Conclusions

Ta/Si (100) and Cu/Ta/Si (100) structures were fabricated by using IBD system with a high purity RF sputter type ion source. We found that applying a bias voltage to the substrate could control the phase of the deposited Ta film, which suggests that ion bombardment plays an important role in the growth of Ta films. Ta films deposited on Si substrates at a bias voltage of -125 V showed non-columnar structure and

had the smallest resistivity of 360 nΩm, which was close to the bulk value of 130 nΩm. The thermal stability measurement of Ta diffusion barrier using Cu(100 nm)/Ta(50 nm)/Si structures showed that the biased Ta film was stable up to 600°C, while the one deposited at zero bias voltage degraded at 300°C. The increase in failure temperature suggests that the stability of Ta films as diffusion barrier layer is dependent on the film microstructure of the deposited layer.

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