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Reliability of multistacked tantalum-based structure as the barrier film in ultralarge-scale integrated metallization

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Abstract

Diffusion barrier properties of Ta films with and without plasma treatments have been investigated in the study. The nitrogen-incorporated Ta films were prepared by NH_3 plasma treatment or reactive sputtering. Barrier properties were evaluated by sheet resistance, X-ray diffraction, transmission electron microscopy, X-ray photoelectron spectroscopy and reverse-biased junction leakage current. An amorphous-like TaN_x layer was formed on Ta barrier film after plasma treatments. The thickness of the amorphous TaN_x layer is about 3 nm and NH_3 plasma-treated Ta films (TaN_x/Ta) possess lower resistivity and smaller grain sizes. The Cu/TaN_x/Ta(10 nm)/Si remained stable after annealing at 750 °C for 1 h. NH_3 plasma-treated Ta films (TaN_x/Ta) possess better thermal stability than Ta and TaN films. It is attributed to the formation of a new amorphous layer on the surface of Ta film after the plasma treatments. For thermal stability of Cu/Ta(-N)/n^+-p diodes, Cu/Ta/n^+-p and Cu/TaN/n^+-p junction diodes resulted in large reverse-bias junction leakage current after annealing at 500 and 525 °C, respectively. On the other hand, TaN_x/Ta diffusion barriers will improve the integrity of Cu/Ta(-N)/n^+-p junction diodes to 650 °C.
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Keywords: Diffusion barrier; Plasma treatment; Thermal stability; Junction diode

1. Introduction

Scaling of ultralarge-scale integrated (ULSI) circuits to critical dimensions 0.5 μm has placed a considerable burden on thin film chip interconnections.

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Continued shrinking of devices has led to a discrepancy between the devices and interconnects performance. The obvious advantages for using Cu as interconnect materials to substitute aluminum alloys are related to improving the operation speed and the reliability of ULSI circuits [1,2]. Compared with aluminum alloys, Cu has a lower bulk resistivity, high electromigration and stress-migration resistance, and higher melting point [3,4]. Recently, Cu has been used for global (long distance) interconnect. It is known that the variation of Cu sheet resistance with anneal temperature provides a good measurement of barrier capability for the Cu/barrier/dielectric system. Furthermore, with continued shrinking of the devices, the local (short distance) interconnects will also change to a Cu metallization system to match the improving performance of ULSI circuits. For the local interconnect, Cu is directly connected to the source/drain area of MOSFETs and the variation of Cu sheet resistance with anneal temperature is no longer a good criterion for evaluating the barrier capability of the Cu/barrier/Si system. However, it is well known that copper diffuses quickly in Si substrate, which causes degradation of transistor reliability by forming particular impurity levels in the silicon [1,2]. Because of its ability to rapidly diffuse in silicon and degrade reliability, the development of effective diffusion barrier materials is the most important issue for the realization of Cu interconnection in Si-based integrated circuits. Many materials were used as a diffusion barrier in copper metallization technique [3–6]. Refractory metals and their nitrides had been investigated for such applications. Among them, tantalum and tantalum nitride have received the most attention owing to their high thermal stability and resistance to form compounds with copper [7–9]. Previous studies have shown that TaN is more desirable than Ta in terms of barrier effectiveness. However, resistivity of tantalum nitride film is higher than that of tantalum film [9,10]. Recently, multilayered TiN/Ti and TaN/Ta barriers as the best barrier for copper metallization were investigated by many researchers [11–13]. As the technology node move to 0.18 μm and below, a thin barrier layer is necessary to lower the resistance of the total line interconnect and/or via. It becomes probably inappropriate to use a multilayered barrier thicker than 30 nm, and hence investigations of the thermal stability and barrier properties of ultrathin barrier layers in the Cu metallization system are important.

In this article, barrier properties and thermal stability of ultrathin Ta-based barrier layers (10 nm) in the Cu metallization system were studied. Furthermore, a new method to form nitrogen-incorporated Ta films with low resistivity and high thermal stability were proposed and investigated. NH_3 plasma was used to post-treat the Ta diffusion barrier. Properties of barrier layers were evaluated by electrical measurements and material analyses.

2. Experimental procedure

Substrates used in the experiment include 6 in. p-type (1 0 0) oriented silicon wafers with resistivity 5–10 Ω cm and oxidized planar (1 0 0) Si wafers and wafers patterned with $1000 \times 1000\ \mu m^2$ n^+-p shallow junction diodes. They were cleaned in a diluted HF solution (HF: H_2O = 1: 50) for 10 min, and rinsed in de-ionized water just before being loaded into a sputtering system. Ta and TaN films, about 10 nm thick, were deposited with no intentional substrate heating and bias by sputtering a Ta target in a dc-magnetron sputtering system (ULVAC SBH-3308 RDE sputter system). Before film deposition, the base pressure of the sputtering chamber was evacuated to approximately 6.67×10^{-5} Pa, and the sputtering pressure was kept at 0.85 Pa by mass flow controllers during sputtering. The pre-sputtering and sputtering power is 250 and 500 W, respectively. The TaN films were prepared using optimum conditions as in our previous investigations, and thus provided a more effective barrier capability against Cu diffusion [9]. Some Ta films were further ex-situ NH_3 treated by the plasma enhanced chemical vapor deposition (PECVD) system (STS Multiplex Cluster system). PECVD chamber is 12 in. The post-treatment temperature is 250°C and plasma power is 250 W. For easy identification, the untreated tantalum, tantalum nitride, and NH_3 plasma treated tantalum films were denoted as Ta, TaN, and TaN_x/Ta, respectively. Afterwards, Cu films

300 nm thick were deposited on top of the Ta(N) films at a power of 1500 W. Cu/barrier/n^+-p junction diodes with LOCOS isolation were used for evaluation of barrier performance. Fig. 1 illustrates schematic diagram of n^+-p junction diodes used in this study. To investigate the barrier capability against Cu diffusion, the diodes were annealed from 400 to 750 °C for 1 h in a vacuum of 1.33 Pa. Leakage current densities of the diodes were measured at the room temperature by a HP4145B semiconductor parameter-analyzer at a reverse bias of −5 V. The sheet resistance of the sample was determined by the four-point probe system. Transmission electron microscopy (TEM) was used for microstructural analyses. Grazing incidence X-ray diffractometry (GIXRD) was carried out for phase identification by Siemens D5000. The incident angle of X-ray was fixed at three degrees and operating voltage and current is 50 kV and 40 mA. The RBS analysis was carried out by using a 2 MeV He^{2+} beam with a backscattered angle of 20°. The elemental compositions of the tantalum nitride film was obtained from RBS data using standard RUMP analysis.

3. Results and discussion

Fig. 2 shows that the variation of normalized resistivity of Ta films before and after plasma treatments. The normalized resistivity is designated as the ratio of R to R_0 (R/R_0), where R_0 and R denote the resistivity of as-sputtered and plasma-treated Ta films, respectively. The resistivity of as-deposited Ta film is 197 μΩ cm. As plasma-treatment time increases from 5 to 15 min, the resistivity slightly increases. However, resistivity of 30-min plasma-treated Ta film increases further. In our previous study, it was found that resistivity of the reactively sputtered TaN film would initially decrease to 159 μΩ cm and then increase to higher than 3500 μΩ cm as nitrogen flow ratio increases during reactive sputtering [9]. Min et al. reported similar results. It was found that small nitrogen incorporation could result in the decrease of the resistivity of reactively sputtered Ta(N) film [15]. The formation of the amorphous layer would increase the resistivity due to increasing scattering effects. The variation in resistivity is due to the combined effects as mentioned above. The nitrogen incorporation is the dominant factor in the early plasma treatment. Resistivity would increase due to the development of the amorphous layer after increasing plasma treatment. It is thought that densification effects could occur due to the bombardments of energetic ions and radicals in plasma treatments. The improvement in the density of the film would result in the enhance-

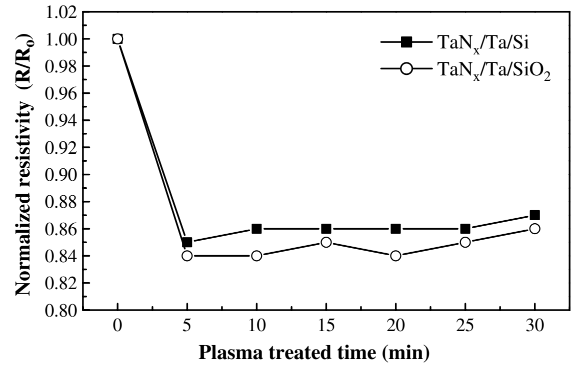

Fig. 2. Normalized resistivity of plasma-treated Ta film as a function of treatment time.

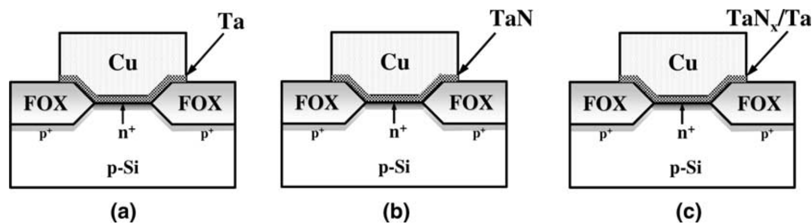

Fig. 1. Schematic diagram of n^+-p junction diodes used in this study.

ment in conductivity of the film. On the other hand, from TEM analyses, the development of an amorphous-like TaN_x layer on the film surface was observed after increasing plasma treatment time, as shown in Fig. 3. The formation of the amorphous layer would increase the resistivity due to increasing scattering effects. The densification effects are the dominant factor in the early plasma treatment. Resistivity would increase because of the development of the amorphous layer after increasing plasma treatment time. It is believed that the variation in resistivity is due to the combined effects as mentioned above.

Fig. 4 shows the X-ray diffraction spectra of tantalum films with NH_3 plasma treatments. The intensity and shape of reflections indicate changes in the microstructure of the tantalum films. As-deposited tantalum films have polycrystalline bcc-Ta structure with (1 1 0) preferred orientation. Plasma treatments result in obvious decrease in the intensities and shapes of reflections of bcc-Ta(N) phases. After the NH_3 plasma treatment for 30 min, the broadening of the peak is obvious, but the crystalline structure is preserved. It reveals that tantalum film becomes nanocrystalline because of bombardments of ions and radicals and surface nitridation.

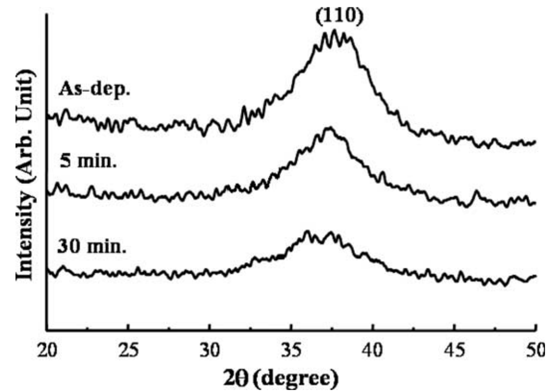

Fig. 4. XRD spectra of the plasma-treated Ta films.

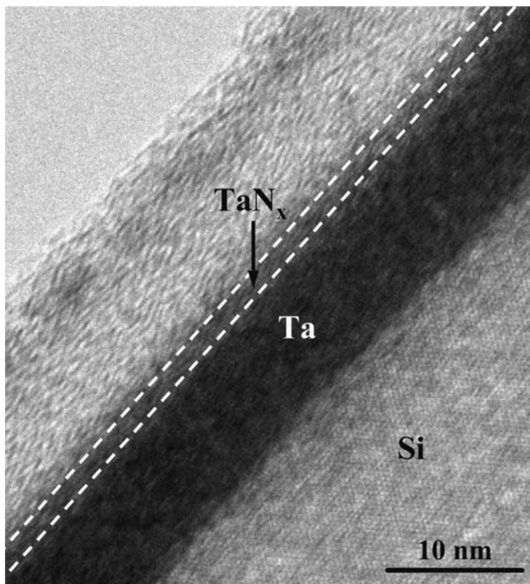

Fig. 3. Cross-sectional TEM micrograph of 30-min plasma-treated Ta/Si.

To further determine the microstructures in the as-deposited and plasma-treated Ta films, the films were analyzed using plan-view TEM. Fig. 5 shows bright field images and selected area diffraction pattern (SADP) of as-deposited and plasma treated Ta films. The bright field image of Ta film clearly shows that the film is bcc-Ta structure with grain size 20–30 nm, as shown in Fig. 5(a). From the results of XRD and TEM, it is concluded that as-deposited tantalum film has bcc structure, which would result in low resistivity. Moreover, the bcc-Ta film has a (1 1 0) texture. Fig. 5(b) shows the TEM image and SADP of Ta with NH_3 plasma treatment for 30 min. It is obvious that the film has an amorphous-like structure. Based on the above investigation, the nitrogen incorporation would contribute to the microstructure transformation from polycrystalline bcc-Ta to amorphous-like TaN_x. It is noted that an amorphous-like TaN_x layer was formed on the surface of Ta film after 30-min plasma treatment. The nitrogen content is about 42 at.%, which detected by RBS. As shown in Fig. 4, it is obvious that as plasma-treated time increases, the (1 1 0) peak of bcc-Ta phase become broad gradually. This is similar to the results of X. Sun et al. [10]. Moreover, with increasing plasma treatment time, the (1 1 0) peak of the film shifts from left to right. The phenomenon indicates lattice distortion and/or

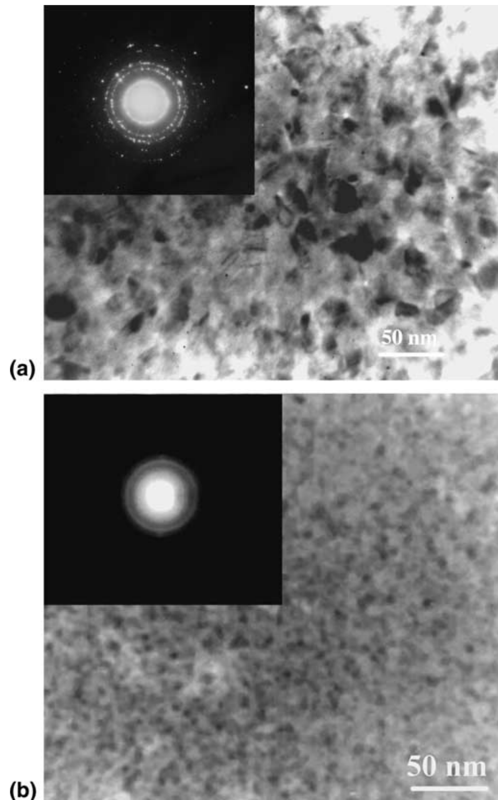

Fig. 5. Plan-view TEM micrographs of (a) as-sputtered Ta film, and (b) TaN_x/Ta.

development of an amorphous-like Ta(N) thin film after plasma treatments. The electron diffraction pattern shows that the plasma-treated Ta film has diffused ring pattern instead of diffraction spots. This indicates that the layer, as shown in Fig. 3, is an amorphous layer. Based on the above investigations, the development of an amorphous layer on the film surface is clearly observed after properly inducing plasma treatment. Several sharp rings are observed for untreated Ta film. It indicates that as-deposited Ta film is polycrystalline structure. The grain size decreases with increasing plasma treatment time. It indicates that nano-crystallization effect would occur due to the reactions and bombardments of energetic radicals and ions during plasma treatment. It is reported that the nanostructured amorphous diffusion barrier, defined as a very short range order single crystal, is highly attractive due to its relatively higher thermal stability against Cu diffusion [19]. Based on the results of microstructure, compositions and resistivity variation, the resistivity increase can be due to extra scattering because of amorphization effect; however, added grain boundaries as the size of the crystallites is getting smaller, due to the nitrogen that is probably at the garin boundaries or due to compoaition change. Hence, the nitrogen incorporation and compoaition change at the grain boundary is the dominant factor in the early plasma treatment. Resistivity would increase due to the development of the amorphous layer after increasing plasma treatment. It is thought that densification effects could occur due to the bombardments of energetic ions and radicals in plasma treatments. The improvement in the density of the film would result in the conductivity variation of the film.

Fig. 6 shows the variation in sheet resistance of Cu/barrier/Si after annealing at various temperatures. The variation in the sheet resistance is designed as the ratio of $(R - R_0)$ to R_0, which R_0 and R denote the sheet resistance before and after annealing, respectively. The results reflect interactions among Cu, barrier and Si substrate. It is noted that the sheet resistance decreases after annealing at 500 °C. It is attributed to a decrease in defect density and grain growth in the Cu film. Sheet resistance of the Cu/Ta/Si sample increased slightly after annealing at 550 °C. After annealing at 600 °C, the color of the sample was found to become gray and the sheet resistance drastically

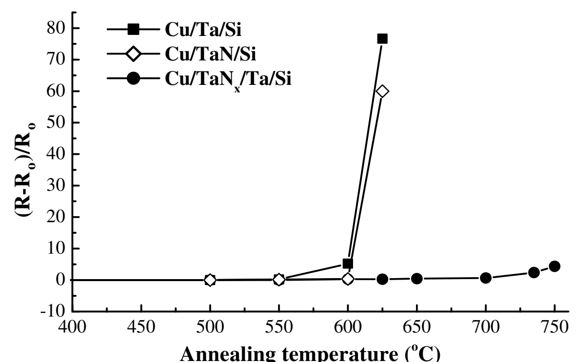

Fig. 6. Sheet resistance of Cu/Ta(-N)/Si as a function of annealing temperature.

increases, indicating that a significant reaction has occurred between the layers. Similar results were found for Cu/TaN/Si annealed at 625 °C. No similar results were found for the Cu/TaN_x/Ta/Si sample even after annealing at 750 °C. It is obvious that NH_3 plasma treated Ta film possesses higher thermal stability than sputtered Ta and TaN films.

XRD analyses identified the phase formation of the Cu/barrier/Si before and after high temperature annealing. Fig. 7(a) shows the XRD spectra of Cu/Ta/Si, Cu/TaN/Si, and Cu/TaN_x/Ta/Si samples without annealing. Strong Cu(1 1 1) and weak Cu(2 0 0) peaks were investigated in all as-deposited samples. It reveals that as copper film was

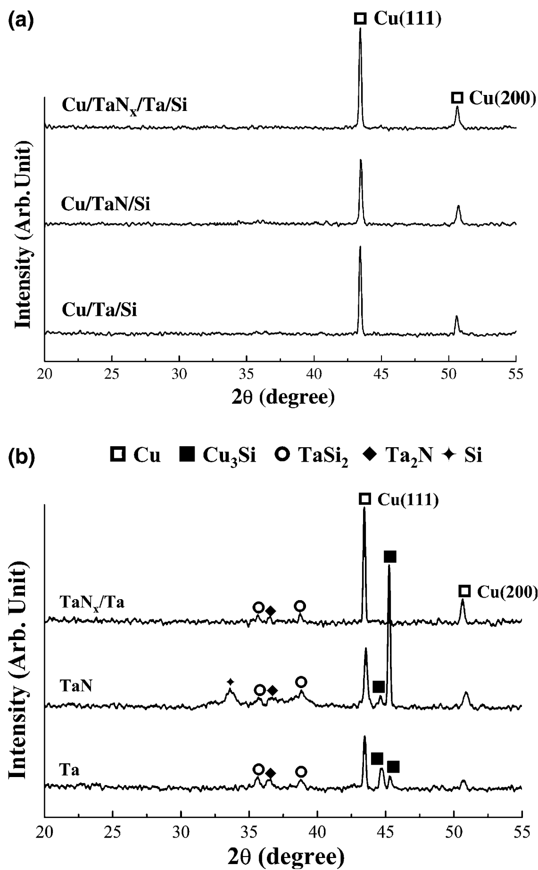

Fig. 7. XRD patterns of Cu/barrier/Si before and after annealing (a) as-deposited Cu/barrier/Si, and (b) XRD spectra of 550-°C annealed Cu/Ta/Si, 625 °C-annealed Cu/TaN/Si, and 700-°C annealed Cu/TaN_x/Ta/Si.

deposited on Ta-based barrier film, copper film possesses (1 1 1)-preferred orientation. Fig. 7(b) shows the XRD spectra of Cu/Ta/Si, Cu/TaN/Si, and Cu/TaN_x/Ta/Si samples after annealing. Strong Cu(1 1 1) and weak Cu(2 0 0) peaks were observed in all annealed samples, indicating that the Cu had preferential ⟨1 1 1⟩ crystal orientation. It had been reported that Cu with high ⟨1 1 1⟩ texture provided higher electromigration resistance [9]. For the Cu/Ta/Si sample annealed at 550 °C, as shown in Fig. 7, signals of $TaSi_2$ and Cu_3Si phases were detected, indicating the failure of the Ta barrier layer. The high-resistivity Cu_3Si formation and related Cu decrease resulted in the great increase of sheet resistance as shown in Fig. 6. For the sample with TaN barrier layer, Cu_3Si phase was also detected after annealing at 625 °C. On the other hand, for Cu/TaN_x/Ta/Si samples annealed at 700 °C, the peak of tantalum silicide was observed, as shown in Fig. 7, whereas copper silicide was not found. It is obvious that 30-min plasma-treated Ta barrier layer can alleviate Cu diffusion into the Si substrate at high temperature and possesses higher barrier stability than sputtered Ta and TaN barrier layers. Fig. 8 shows the cross-sectional TEM micrographs of Cu/TaN_x/Ta/Si and TaN_x/Ta/Si after annealing and copper was stripped by HNO_3 solution, respectively. Fig. 8(a) shows cross-sectional TEM micrographs of Cu/TaN_x/Ta/Si systems after annealing at 700 °C for 1 h. The multilayered structure is obvious. That is, the multilayered structure Cu/TaN_x/Ta/Si is retained. No Cu silicides are observed at the interface, demonstrating the excellent barrier properties. Fig. 8(b) shows cross-sectional TEM micrograph of TaN_x/Ta/Si after annealing at 700 °C. As shown in Fig. 8(b), tantalum silicide was observed on the TaN_x/Ta and Si interface, indicating an interaction between barrier and silicon. However, the amorphous TaN_x layer was existed obviously even though annealing at high temperature. Furthermore, no copper silicide was formed in silicon substrate. These copper spiking was presumably caused by Cu diffusion through the localized weak points in the barrier and reacting with underlying Si to form Cu_3Si. It is believed that the amorphous TaN_x layer possesses better capability against copper diffusion.

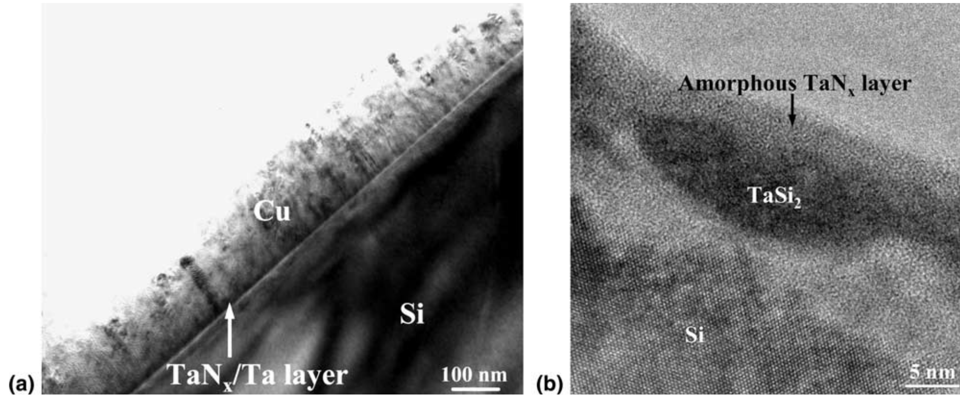

Fig. 8. Cross-sectional TEM micrographs of (a) Cu/TaN_x/Ta/Si, and (b) TaN_x/Ta/Si after 700 °C annealing and copper was stripped by HNO_3 solution, respectively.

For the Cu contacted interconnect system, it is known that the leakage of the diode is more sensitive to the barrier degradation than the variation in the sheet resistance [9]. Fig. 9 illustrates the plots of reverse-biased leakage current densities measured at −5 V for Cu/Ta/n^+-p, Cu/TaN/n^+-p and Cu/TaN_x/Ta/n^+-p junction diodes annealed at various temperatures. If we define a failure criterion with 10^{-6} A/cm^2, the Cu/Ta/n^+-p junction diodes are stable under thermal annealing at temperature up to 475 °C, but suffer degradation at 500 °C. For the diodes with 10 nm TaN barriers, the diodes remained stable after annealing at temperature up to 500 °C. After annealing at 525 °C, failure of the diodes was observed. Similar results were reported by Tsai et al. It was found that the diodes with 60 nm CVD and PVD TaN barriers would begin to deteriorate at 500 and 550 °C, respectively [14,15]. It is shown that barrier capability of TaN film is better than that of Ta film. However, the improvement is limited. Furthermore, the resistivity of reactively sputtered TaN will significantly increase with increasing nitrogen concentration. In our previous study, it was found that resistivity of the TaN film sputtered at 35% nitrogen flow ratio would increase to ∼3500 from ∼190 μΩ cm for the Ta film [9]. As mentioned previously, nitrogen-incorporated Ta films (TaN_x/Ta) were also prepared by ammonia plasma treatments in the study. The resistivity of TaN_x/Ta was as low as that of as-sputtered Ta, as shown in Fig. 2. The Cu/TaN_x/Ta/n^+-p diodes retained integrity even after annealing at 650 °C, as shown in Fig. 9. The TaN_x/Ta films possess much better barrier performance than sputtered Ta and TaN films. It is believed that improved barrier performance is attributed to nano-crystallization and stuffing effects due to the reactions or bombardments of energetic radicals and ions during plasma treatments. It is reported that the microstructure within the barrier layer strongly affects the barrier performance because it is believed that Cu diffuses through fast diffusion path such as grain boundaries within the barrier layer [9,16–18]. The thin nano-crystalline TaN_x layer was formed on the surface of the Ta film after ammonia plasma

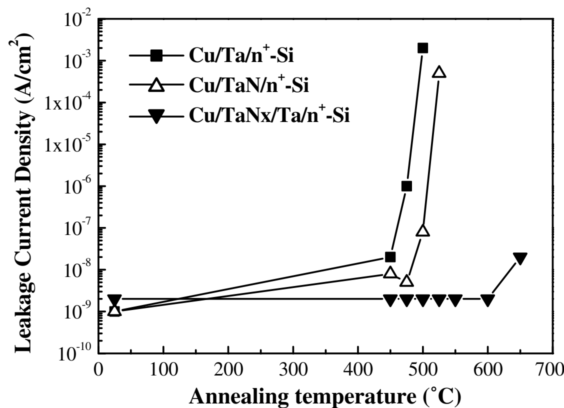

Fig. 9. The plots of junction leakage current densities of Cu/Ta/Si, Cu/TaN/Si, and Cu/TaN_x/Ta/Si junction diodes after annealing at various temperatures for 1 h.

treatments, as shown in Fig. 3. Nanostructured diffusion barrier is more effective for preventing Cu diffusion than the polycrystalline barrier since the nanostructured film does not have large-angle grain boundaries where most of the atomic diffusion generally occurs [18].

4. Conclusion

In this study, the barrier capability of the sputtered Ta, TaN and Ta film with NH_3 plasma treatment against Cu diffusion was investigated. It is found that Cu/Ta/n^+-p and Cu/TaN/n^+-p junction diodes remain stable at a temperature up to 475, and 500 °C, respectively. Cu/Ta/n^+-p and Cu/TaN/n^+-p junction diodes result in large junction leakage current after annealing at 500 and 525 °C, respectively. Plasma-treated Ta films will improve the integrity of Cu/Ta/n^+-p junction diodes to 650 °C. It is attributed to the formation of a new amorphous TaN_x layer on the surface of Ta film. Nanostructured diffusion barrier is more effective for preventing Cu diffusion than the polycrystalline barrier since it does not have large-angle grain boundaries where most of the atomic diffusion generally occurs. The plasma treatment provides a new method of forming nitrogen-incorporated Ta films with low resistivity, small grain size, and high thermal stability.

Acknowledgments

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