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The impact of layer thickness of IMP-deposited tantalum nitride films on integrity of Cu/TaN/SiO_2/Si multilayer structure

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Abstract

This work concentrates on the diffusion barrier stability of very thin tantalum nitride films with different thickness (10, 20 and 30 nm) sputter-deposited on silicon dioxide in the Cu/TaN/SiO_2/Si multiplayer structure. The impact of varying layer thickness and influence of post deposition annealing on the crystal structure, resistivity, intermixing and reactions at the interfaces were studied by using resistivity analyses, X-ray diffraction, scanning electron microscopy and Rutherford backscattering spectrometry. The results revealed that the thinner the film thickness of tantalum nitride, the more severe the reactions at the interface of copper–tantalum nitride and consumed more conductive Cu. Accelerated grain growth and/or agglomerations were also observed in all Cu surfaces. All the structures show a similar degradation process and were found to be stable up to 450°C for 35 min.

Keywords: Tantalum nitride (TaN); Ionized metal plasma (IMP); Diffusion barrier

1. Introduction

Current semiconductor technology dictates the use of low-resistivity metal lines for multilayer interconnection devices. Currently, Al–Cu metal layers are employed in most devices; however, copper has emerged as an attractive alternative. This trend is reinforced by the fact that Cu offers lower resistivity and superior electromigration and stress-voiding resistance over Al and its alloys [1]. However, several Cu-related problems, including high mobility, strong reactivity and poor adhesion, should be solved first in order to fully integrate Cu into integrated circuits. However, the successful implementation of Cu-based metallization for ULSI Si devices requires not only new manufacturing technologies and design modifications but also new material systems that act as effective diffusion barriers, protection layers and adhesion promoters of low-k dielectric.

Electrical and structural properties of tantalum (Ta) thin films have received considerable attention owing to their widespread applications in electronic devices and X-ray lithography [2–4]. Since the advent of Cu interconnects for deep submicron multilevel integrated circuits, Ta is a highly promising polish stop and adhesion layer for chemical mechanical polishing (CMP) of the damascence process [5]. Furthermore, as generally accepted, Ta and nitrogen-contained tantalum (Ta–N) thin films are the most promising diffusion barriers to prevent the highly diffusing Cu from reacting with the underlying silicon and surrounding SiO_2 dielectric [6–12]. Thus, the feasibility of growing Ta and TaN thin film has been extensively studied, particularly in terms of controlling the phase, electrical property and microstructure of the Ta and TaN films so that they can be used as reliable barriers between Cu and Si/SiO_2 [6–12]. In particular, a reliable and high conductive thin-film diffusion barrier of resistivity less than 300 $\mu\Omega$ cm^{-1} and thickness less than 40 nm should be employed to effectively retard Cu from intermixing and reacting with Si. Indeed, the ability of a wide variety of transition metal-related thin film barrier materials for Cu has been extensively investigated. According to the relationship between critical dimension and barrier thickness, for the dimensions below 0.18 μm the barrier thickness should be reduced down to 10 nm in order to

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achieve a gain in performance. Additionally, both increasing the aspect ratio of the vias and decreasing their cross-section cause barrier thinning at via sidewalls and bottoms. The necessary reduction in barrier thickness places severe requirements on the barrier quality. In our present work, the TaN diffusion barrier with different thicknesses were deposited by ionized metal plasma (IMP) sputtering. It will overcome the generic PVD processing limitations such as the poor step coverage without losing the excellent metallurgical diffusion barrier properties. In addition, IMP deposition makes the TaN film microstructure denser, in particular, in grain boundaries. Hence, the diffusion and intermixing of Cu through TaN film can be blocked effectively due to the discontinuous grain boundaries, which is expected to improve the barrier properties much better than reactive sputtered TaN. The test sample structure is Cu (200 nm)/TaN (10, 20, 30 nm)/SiO_2 (500 nm)/Si and the diffusion barrier properties were evaluated by electrical measurement. Furthermore, XRD, SEM and RBS were employed in conjunction with electrical measurements to examine the failure mechanism.

2. Experimental details

For all sample preparation and experiments described in this study we used 8-inch Si (100) wafers. Si wafers were cleaned in 10:1 diluted HF solution and rinsed in deionized water before SiO_2 deposition. First, a 500-nm thick plasma enhanced chemical vapor deposited (PECVD) SiO_2 dielectric was deposited by using a gas mixture of SiH_4, Ar and O_2 at 400°C on 8-inch Si wafers. Tantalum nitride (TaN) films of different thickness, 30 nm (sample A), 20 nm (sample B) and 10 nm (sample C) which act as a diffusion barrier and adhesion layer for the highly conductive Cu atoms, were deposited onto PECVD-SiO_2 (500 nm)/Si substrates by using ionized metal plasma (IMP) sputtering in a gas mixture of Ar and N_2. Without breaking the vacuum, a 200-nm Cu layer was then deposited by IMP sputtering. A detailed IMP deposition process has been described elsewhere [13].

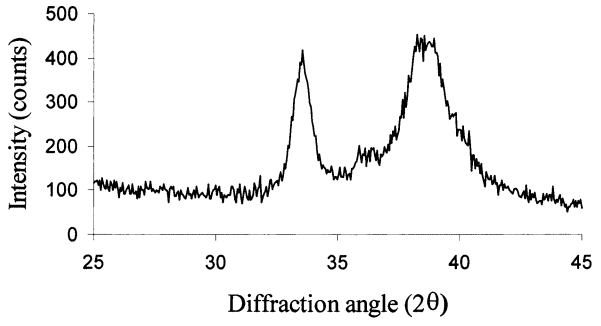

Fig. 1. XRD pattern of as-deposited TaN showing amorphous and/or nanocrystalline structure.

3. Results and discussion

Fig. 1 shows the XRD scan result for as-deposited TaN films used in this experiment. It can be clearly seen that TaN film is composed of very fine grains (nanostructure and/or amorphous) since diffraction pattern of the film contains two broad low intensity peaks having a full width at half maximum of 6° (second one). This observation implied that this TaN film is constituted mainly of an amorphous phase material. Notably, two different phases, such as amorphous and crystalline Ta_2N, or Ta_2N and TaN, can coexist within the limited range of N_2 flow rates [11,14]. Regardless of the thickness the microstructure of as-deposited tantalum nitride (sample A, B, and C) films were found to be the same. The surface morphology was examined by atomic force microscopy. AFM was performed using a Nanoscope III multimode atomic force microscope. Data were collected in tapping mode AFM with silicon cantilevers at resonance frequencies in the range of 200–300 kHz. Fig. 2 shows the AFM measurement results of the as-deposited tantalum nitride films with different thicknesses. It was observed that the lower the thickness the smoother the surface of the samples since roughness (RMS) of sample A (30 nm TaN) is approximately ~ 1.714 nm, sample B (20 nm TaN) is approximately ~ 1.645 nm and sample C (10 nm TaN) is approximately ~ 1.462 nm. The resistivity of the as-prepared tantalum nitride films was also examined by measuring the sheet resistance using the four-point probes method. The as-deposited films (sample A, B, and C) show a value of ~ 200 $\mu\Omega$ cm^{-1}, which agrees well with the minimum value of the resistivity of the TaN compound reported by Hieber [15].

The graphs presented in Fig. 3 indicate the change in sheet resistance measured on the Cu/TaN/SiO_2/Si structure as a function of annealing temperature in N_2 ambient for 35 min. The measured sheet resistance was dominated by the Cu thin film since the copper film (200 nm and 1.72 $\mu\Omega$ cm^{-1}) is much thicker and has a markedly lower resistivity than that of tantalum nitride films (10, 20, 30 nm and ~ 216 $\mu\Omega$ cm^{-1}) and any reaction products. Since the top Cu layer of 200 nm carries nearly all the sensor current, the sheet resistance measurements monitor the condition and the quality of the Cu overlayer. Hence, theses curves can be used to estimate the degree of intermixing, reaction, and changes of integrity across the metallization layers as well. According to this figure, all samples, annealed up to 450°C can maintain the same level of sheet resistance as the as-deposited samples. However, the sheet resistance increases slightly at temperatures just exceeding 450°C, implying that a relatively resistive substance has been produced from the highly conductive Cu layers. As the temperature reaches 550°C and beyond, the sheet resistance of the samples rises abruptly, indicating

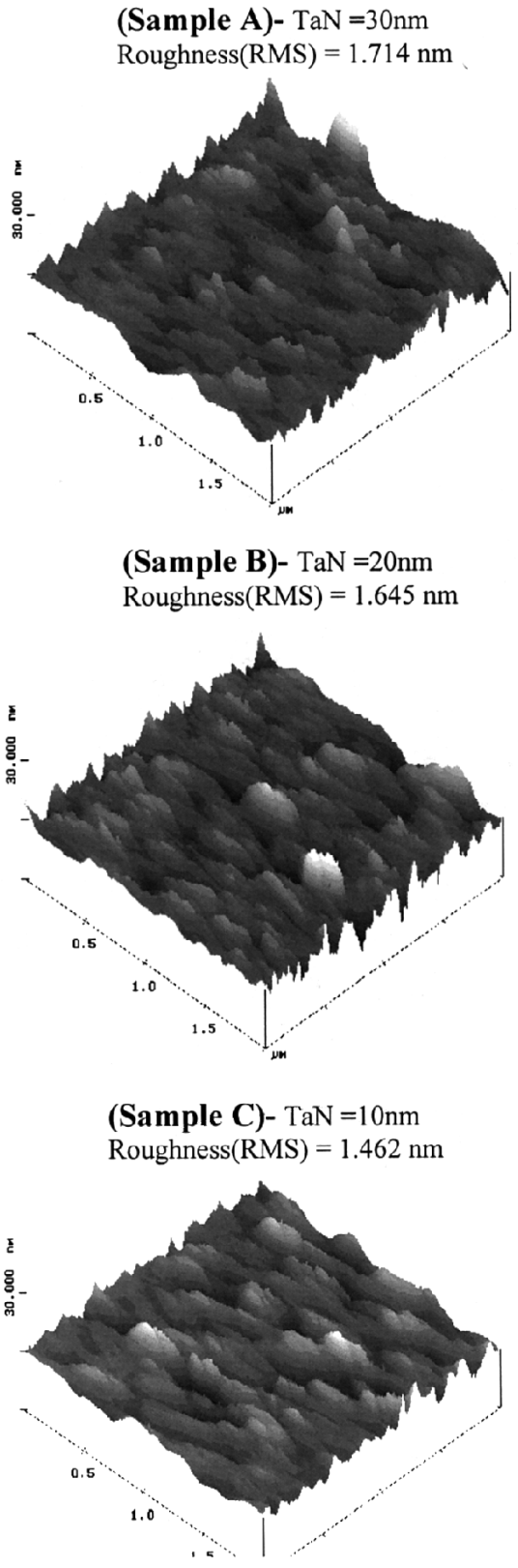

Fig. 2. AFM measurement results of the as-deposited TaN samples with different thicknesses.

that a severe intermixing or interfacial reactions occurred across all the Cu films.

To identify the new phases formed during the annealing, X-ray diffraction analysis (XRD) was carried out to evaluate the interaction between layers. XRD measurements were performed with the grazing incident angle (2.5°) attachment in a RIGAKU RINT-2000 diffractometer, using Cu K_α X-ray at 50 kV and 20 mA from 10° to 120° with a 0.05° step and 1°/min scanning rate.

Fig. 4 displays a typical series of XRD patterns for freshly prepared Cu/TaN/SiO_2/Si (sample A) structures and annealed at temperatures ranging from 350 to 650°C. The diffraction pattern reveals that the as-deposited tantalum nitride thin films indeed exhibit two broad low intensity peaks. Subsequently, after annealing 350°C and 450°C, these broad peaks are converted into a single sharp peak that reach saturated intensities and locate at the position corresponding to hexagonal Ta_2N.[1] This transformation suggests that the sputter-

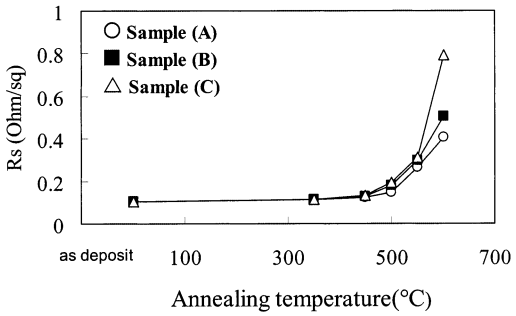

Fig. 3. Variation of sheet resistances of Cu/TaN/SiO_2/Si multilayer structures as a function of annealing temperatures.

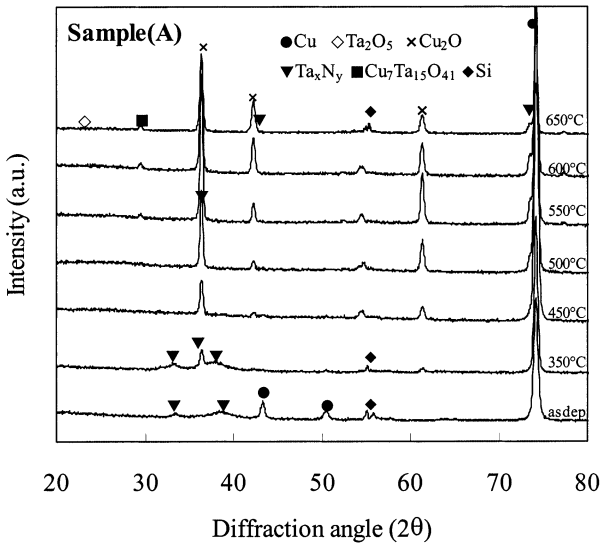

Fig. 4. XRD patterns of the Cu/TaN (30 nm)/SiO_2/Si multilayer structure annealed at various temperatures for 35 min in N_2 ambient.

[1] JCPDF cards no. Cu (040836), Cu_2O (050667), Ta_2O_5 (181304), Ta_2N (260985), and $Cu_7Ta_{15}O_{41}$ (410208).

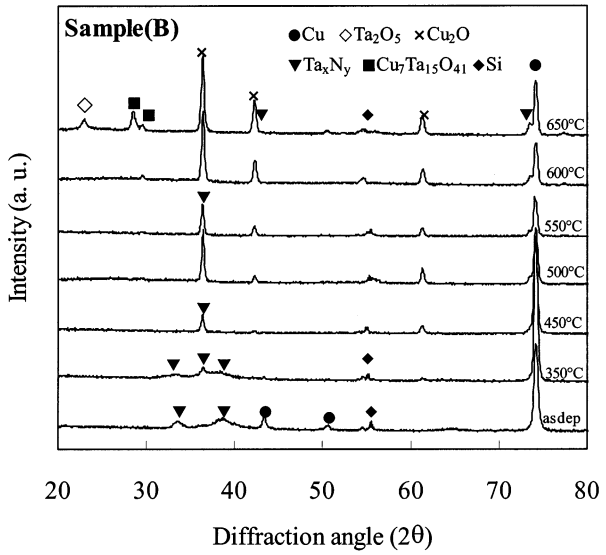

Fig. 5. XRD patterns of the Cu/TaN(20 nm)/SiO_2/Si multilayer structure annealed at various temperatures for 35 min in N_2 ambient.

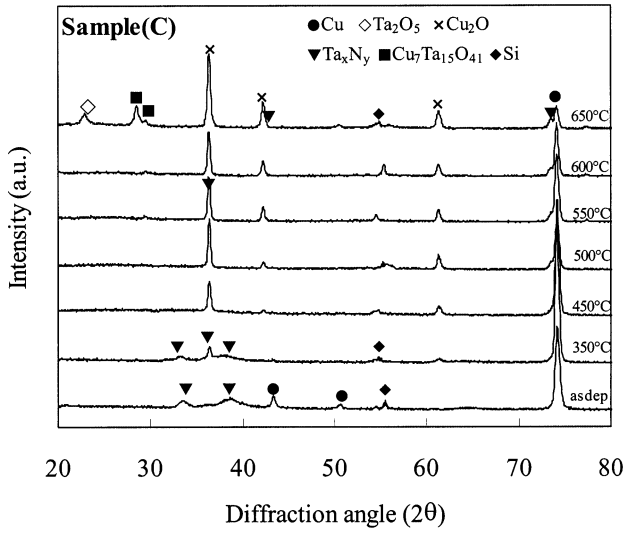

Fig. 6. XRD patterns of the Cu/TaN(10 nm)/SiO_2/Si multilayer structure annealed at various temperatures for 35 min in N_2 ambient.

deposited tantalum nitride diffusion barriers are mainly amorphous (a mixture of amorphous and crystalline Ta_2N), and can easily undergo a crystallization transition at temperatures as low as 450°C. This peak was overlapped by the Cu_2O peak, which starts forming at 450°C annealing. The intensities of the Cu (220) peak increase after annealing 350°C, presumably due to the annihilation of the inherent crystal defects and grain growth of the Cu layer. However, the intensities of this peak slightly decrease again as the annealing temperature surpasses 500°C. This suggests that some Cu atoms were consumed by forming Cu_2O. But the formation of Cu_2O at 2θ angles 36.35°(111), 42.36°(200) and 61.30°(220) become prominent as annealing temperature increase. After 650°C annealing, two peaks with very low intensity were found at 22.8° and 28.35° and they are identified as Ta_2O_5 and $Cu_7Ta_{15}O_{41}$.

Another series of XRD patterns presented in Fig. 5 indicate that the Cu/TaN/SiO_2/Si (sample B) structure follow a transition process similar to that of sample A. However, the intensities of Cu (220) peak decrease continuously as the annealing temperature surpasses 500°C. The intensity of the Cu peak after 650°C was found to be very much less than that of in sample A and the peaks of new compounds were more visible compared to sample A. The X-ray diffraction pattern display in Fig. 6 clearly reveals that the Cu peak (of sample C) after annealing at 650°C was significantly reduced and Ta_2O_5 and $Cu_7Ta_{15}O_{41}$ peaks were stronger in intensity compared to both sample A and B. This observation leads to a conclusion that the thinner the Ta_2N barrier the more severe the reactions between Cu and Ta_2N and consumed a more conductive Cu layer by forming $Cu_7Ta_{15}O_{41}$.

The mechanisms of Cu film oxidation at low temperatures were studied by Li et al. [16]. It was observed that Cu is first oxidized to Cu_2O at temperatures as low as 200°C. Since the samples used in this study were protected by the inert N_2 ambient during annealing process the formation temperature of Cu_2O increased significantly and no formation of CuO was observed. In the case of Ta_2O_5, according to the Ta–O binary system, up to approximately 5 at.% of the oxygen could be interstitially dissolved in the b.c.c-Ta lattice prior to conversion into the amorphous or polycrystalline phase [17]. However, in our structure the as-deposited tantalum nitride diffusion barrier is mainly an amorphous phase (a-TaN). It is thought that, the a-TaN then transits from the amorphous phase to a mixture of TaN and Ta_2N upon thermal annealing (does not alter the resistivity of the film). However, Ta_2N is unstable and dissociates into body center cubic (α-phase) Ta and a Ta_2N phase [14]. On the other hand, Ta has very high affinity to oxygen and reacts with it and forms Ta_2O_5. As impurity in polycrystalline TaN film, O is believed to increase the effectiveness of the diffusion barrier by decorating the extended defects such as grain boundaries, thereby blocking the active paths for grain boundary diffusion [7].

Here, three sources of oxygen atoms forming Cu_2O, Ta_2O_5 and $Cu_7Ta_{15}O_{41}$ in all samples will be considered. Firstly, oxygen atoms from the SiO_2, which would diffuse into and/or react with the Ta_2N layer, as the annealing temperature increased since Lane et al. [18] reported the possible reactions between SiO_2 and TaN and the formation of Ta_2O_5 at the interface of TaN/SiO_2. The second source is oxygen atoms incorporated with Cu and Ta metal film from the deposition ambient during the Cu and TaN deposition and decorating the grain boundaries of each film [19]. We also reported the

oxygen and carbon concentration and the depth profile in IMP-TaN and IMP-Cu, being examined by SIMS analysis and the formation of Cu_2O and Ta_2O_5 [20]. Lastly, oxygen atoms incorporated from annealing ambient, but were excluded because all the samples were annealed in an 'inert' nitrogen atmosphere. However, the formation of Cu_2O and/or Ta_2O_5 cannot be fully prevented. This reveals the fact that formation of Cu_2O, Ta_2O_5 and $Cu_7Ta_{15}O_{41}$ in the structure is mainly due to the oxygen incorporated during the deposition process. The intensity of Cu (200) peak was slightly reduced in all samples due to the formation of Cu_2O. Annealing at temperatures higher than 450°C makes Cu and the

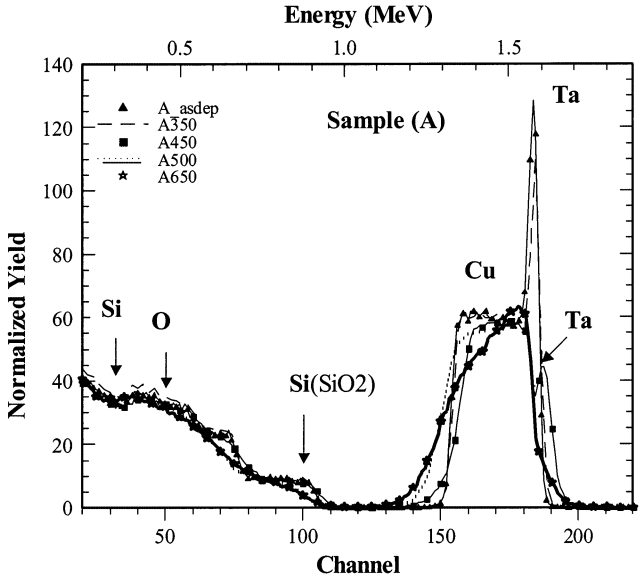

Fig. 7. 2 MeV He^+ RBS spectra of Cu/TaN(30 nm)/SiO_2/Si multilayer structure annealed at various temperatures.

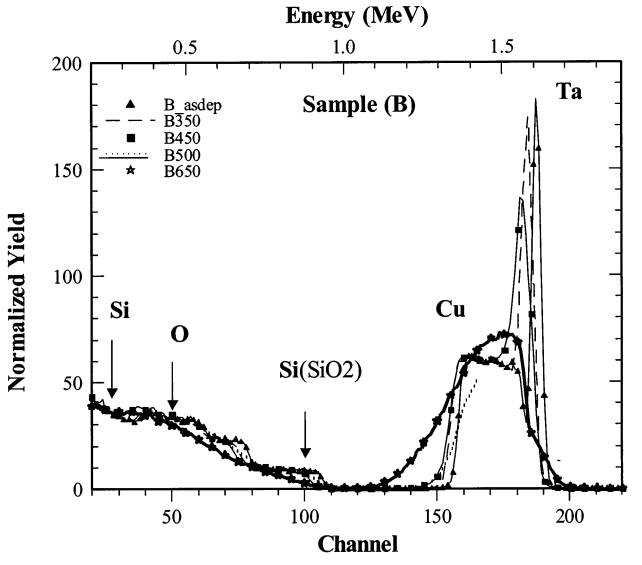

Fig. 8. 2 MeV He^+ RBS spectra of Cu/TaN(20 nm)/SiO_2/Si multilayer structure annealed at various temperatures.

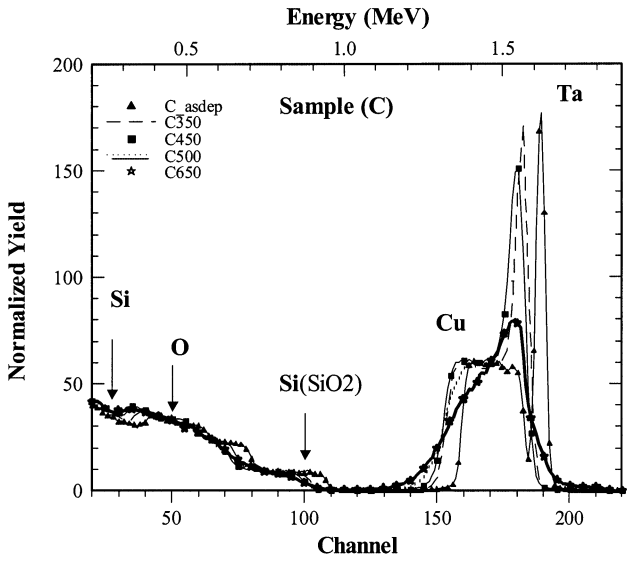

Fig. 9. 2 MeV He^+ RBS spectra of Cu/TaN(10 nm)/SiO_2/Si multilayer structure annealed at various temperatures.

dissociated b.c.c.-Ta start to react with the O_2 existing in the grain boundaries of Cu as well as Ta_2N resulted in the formation of Cu_2O and Ta_2O_5. By annealing at 650°C, a new peak of $Cu_7Ta_{15}O_{41}$ appeared at 28.35°, probably due to the reaction among Cu_2O, Ta_2O_5, Ta and Cu at the interface of Cu/Ta_2N [21]. As a result, a reduction in intensity of Cu (220) and Ta_2N peaks was also observed due to the formation of Cu_2O, Ta_2O_5, and $Cu_7Ta_{15}O_{41}$. The peak of a new compound was observed very close to the Ta_2O_5 peak. Although an interfacial reaction and the formation of Cu_2O, Ta_2O_5 occurred in all structures, no evidence of the diffusion of Cu through the barrier (copper silicide formation) was detected even after annealing at 650°C for 35 min, which indicates that the Ta_2N diffusion barrier layer effectively suppresses the reaction between the Si and Cu overlayer.

Rutherford backscattering spectrometry (RBS) spectra were taken with 2 MeV He^+ ions at a scattering angle of 160° using a 50-mm^2 passivated implanted planar silicon (PIPS) detector of 13.5 keV resolution. Figs. 7–9 show the RBS spectra of Cu/TaN/SiO_2/Si structures annealed at various temperatures for 35 min. The surface energies of Cu and Ta have been indicated. Rutherford backscattering (RBS) measurements were performed to evaluate the interaction between the different layers. Fig. 7 shows the RBS spectra of sample A annealed from 350 to 650°C for 35 min. At 350°C, the RBS spectrum shows sharp layer structures. At a higher annealing temperature of 450°C, the Ta peak position for sample A shifts from 1.57 MeV to a higher energy at 1.60 MeV. This observation suggests either an out diffusion of Ta into the Cu film or a reduction in the Cu thickness. Nevertheless, the Cu surface seems to be

free of Ta atoms since the energy of the Ta peak observed is lower than the Ta surface peak energy. It seems that the out diffusion of Ta atoms is mainly distributed within the Cu film grain boundaries. At 500°C, the Ta peaks were observed to have broadened and moved back to a lower energy. Tailing of the Cu peaks was observed at the low energy edge, which suggest the occurrence of agglomeration in the Cu layer. This is also confirmed by the SEM micrographs of the structure after annealing at 600 and 650°C (Fig. 10). The lower Cu yield suggests the formation of Cu oxide at the surface. The XRD result shown in Figs. 4–6 also supported this oxide formation. The composition of the oxide is determined to be Cu_2O from the XRD result. The presence of the surface Cu oxide for the sample annealed at 500°C could explain the increase in the sheet resistance by $\sim 200\%$.

Fig. 8 shows the RBS spectra for sample B (with 20-nm thick Ta) annealed from 350 to 650°C for 35 min. The Cu concentration yield increases as the annealing temperature increases from 350 to 450°C. This agrees well with the XRD results, which show an increase in intensity of the Cu (220) peak. As the Cu layer thickened, presumably due to grain growth of the Cu layer, the Ta peak also shifts to lower energy. After 500°C annealing, a slight reduction in the Cu yield was observed which indicates the formation of the surface Cu oxide. For even higher annealing temperatures, the Ta peak position is shifted back to a lower energy and broadening of the Ta and the Cu peaks were observed. The higher sheet resistance measured at 550°C could be attributed to the loss of the conductive Cu layer. The formation of surface Cu_2O could result in this loss of the conductive Cu layer. The following features were observed for 650°C annealing: (i) a significant reduction of the Ta peak height and the broadening of the Ta peak; and (ii) the tailing of the Cu peak into even lower energy. These features indicate a possible formation of a new Cu–Ta compound. The XRD result, shown in Figs. 4–6, revealed the formation of $Cu_7Ta_{15}O_{41}$ and a

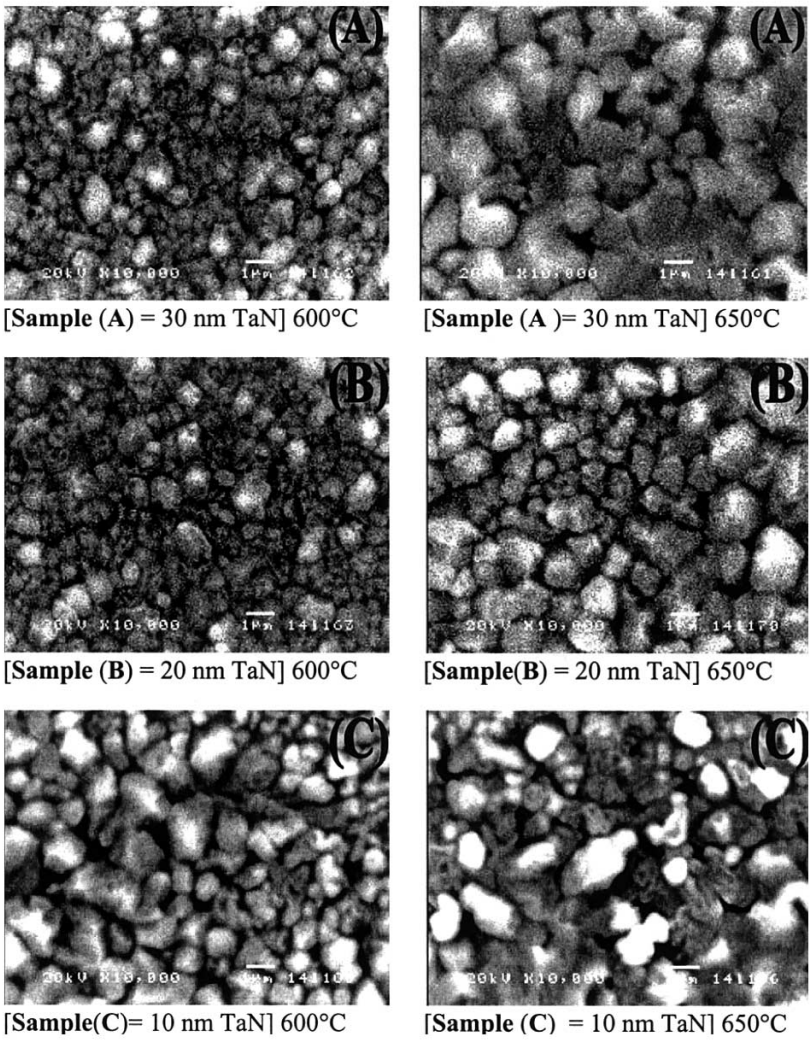

Fig. 10. SEM micrograph of the Cu/TaN/SiO_2/Si multilayer structures after annealing at 600 and 650°C.

significant reduction of the Cu peak. The RBS spectra for sample C given in Fig. 9 shows very similar phenomena to that for sample A and B. However, the Cu yield annealed at 650°C shows a significant reduction, again this agrees well with the XRD results which show a very low Cu peak intensity (Fig. 6) at 650°C. This extensive loss of conductive Cu layer, thus giving the highest increase in sheet resistance value is shown in Fig. 3.

Fig. 10 shows the surface morphologies of Cu film after annealing at 600 and 650°C in N_2 ambient for 35 min. The average grain size of an as-deposited Cu film was approximately 80 nm and it grew as annealing temperature increased but grain growth occurs uniformly. Therefore, it is better described as normal grain growth. Besides grain boundary energy reduction, surface energy and strain energy reductions are the driving forces for grain growth in thin films [22,23]. Due to the thin oxide (copper oxides) formed on Cu surfaces, the surface energy of Cu grains or the stress of the film will change, and these variations may cause the accelerated grain growth. It was reported that normal grain growth occurs until the nominal grain size of the film becomes two to four times larger than the film thickness, while abnormal grain growth gives a preferential growth of some grains [24,25]. Zielinski et al. [26] reported that if surface energy and stress variation were significantly different with different orientations of grains, some specially oriented grains would grow abnormally to reduce the total system energy. Conversely, if surface energy and stress variation are uniform with the orientation of the grains, abnormal grain growth will not be observed. In our present work, no evidence of abnormal grain growth in Cu film was observed during thermal annealing. As shown in Figs. 4–6 a (220) preferred orientation was maintained throughout the annealing process, and no abnormal grains were observed in SEM images. These facts reveal that normal grain growth occurs in this Cu film during the annealing process. However, it is not likely that the uniform variation of surface energy with grain orientation induces the grain growth in thin films [26]. Since the volume of grain boundaries reduces during the grain growth, tensile stress is produced in film. As a consequence, only a compressive stress condition can be relaxed by grain growth [26,27]. Halliday et al. also reported that the oxidation of Cu increased the compressive stress in the film due to the formation of a superficial oxide layer [28]. This suggests the fact that compressive stress induced from the oxidation of Cu becomes the driving forces for the normal grain growth of Cu thin film in our structures.

4. Conclusions

This work investigated the impact of the difference thickness of the TaN diffusion barrier in the Cu/TaN/SiO_2/Si multiplayer structure. Results obtained from high temperatures annealing of the samples pointed out that the property of any thickness of diffusion barriers is mainly deteriorated by their crystallization and out diffusion of Ta towards the Cu layer. The TaN layer having an optimal thickness (10 nm) can maintain its integrity the same as the higher thickness one (20 and 30 nm).

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