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A Study on the Diffuse Mechanism and the Barrier Property of Copper Manganese Alloy on Tantalum

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ABSTRACT In this paper, the electrical and material properties of CuMn/silicon oxide (SiO_2) and CuMn/tantalum (Ta)/SiO_2 were investigated, and an optimized concentration of Mn in the CuMn alloy as barrier layers in these two structures was also determined. CuMn alloy (0∼10 atomic % Mn) deposited on SiO_2 and Ta were used in this paper. A diffusion barrier layer self-formed at the interface during annealing, and the growth behavior was found to follow a logarithmic rate law. The microstructures of the CuMn films were analyzed by transmission electron microscopy and could be correlated with the electrical properties of the CuMn films. After thermal treatment, only Cu-5 at.% Mn/ SiO_2 successfully avoided the diffusion of Cu atoms. Thermal stability of the films grown on Ta/SiO_2 was found to be better than that on SiO_2. When a Ta layer was added, the Mn atoms diffused not only to the interface, but also to the grain boundaries in the Ta layer and the interface between Ta and SiO_2. This phenomenon could be explained by the surface energy. As the thickness of CuMn shrunk from 150 to 50 nm and the sample was covered with a 100-nm-thick Cu layer, the amount of Mn atoms increased at the interface of CuMn/Ta. This is because the Cu layer had higher chemical potential which induced the Mn atoms to move toward the Ta layer and reduced the amount of Mn atoms in Cu after heat treatment.

INDEX TERMS Barrier, self-formed, annealing, CuMn alloy, Ta.

I. INTRODUCTION

In sub-130 nm semiconductor device manufacturing processes, copper (Cu) has been used as the interconnection metal as replacement for aluminum (Al) because Cu metal has higher electromigration resistance and lower resistivity. The double layer structure of tantalum (Ta) / tantalum nitride (TaN) are called a barrier layer as a single entity and are necessary in metallization process to prevent interdiffusion between Cu and Si atoms. As the minimum feature size of microelectronic devices shrinks down to 32 nm, 22 nm and beyond, one challenge in semiconductor manufacturing is the increase in resistance of metal lines with feature shrinkage. Previous researchers have reported an alternative to the conventional barrier process called "self-forming" barrier process. This process involves the deposition of a Cu alloy thin film directly on SiO_2 followed by heat treatment to trigger the migration of the alloying element to the alloy/SiO_2 interface and to form a thin barrier layer via reaction with SiO_2 [1]–[8]. However, previous investigations found that it was difficult to form a thin barrier layer and to reduce the resistance of the metal lines simultaneously.

Recently, CuMn alloy process was proposed by Koike and Wada [9] and Iijima *et al.* [10]. An excellent barrier layer self-formed by annealing the CuMn alloy at 450 °C. Usui *et al.* [11] reported that a self-forming barrier layer can form without the conventional Ta barrier and also demonstrate excellent resistance against stress-induced voiding and electromigration failure. The growth behavior and thermal stability of the self-forming barrier layer were also reported [12]. The Mn concentration is one of the key factors in this process. Some articles mentioned that the Mn concentration affects the thermal stability of the barriers [13].

The thermal stability, thickness of the barrier and the composition of the metal layer are sensitive to the Mn concentration, temperature and time of the annealing.

In this paper [14], we used a CuMn/Ta stack layer as the barrier. The diffusion behavior of the metal atoms during heat treatment and barrier properties with different Mn concentration in the alloy from 0% to 10% were reported. The diffusion mechanism of the Mn atoms between CuMn and Ta, thermal stability, microstructure, and sheet resistance are also discussed.

II. EXPERIMENTAL DETAILS

A SiO_2/p-Si (111) wafer was chosen as the substrates for this study. The 400-nm-thick SiO_2 layer was grown on the Si substrate by thermal oxidation. The metal layer of 99.99% purity was sputtered onto SiO_2/Si ($\sim 2 \times 2$ cm^2) at room temperature by direct-current magnetron-sputtering with the Ta target and the Cu-0$\sim$ 10 at.% Mn alloy targets and the diameter of each target is 2 inches. The thickness of Ta and CuMn was controlled at 28 nm and 150 nm, respectively. The sputtering power was kept at 20 W, and the base pressure and working pressure were controlled at 10^{-6} Torr and 5 mTorr, respectively. Before sputtering, the sample was degassed at 200 °C for 60 s. Deposition was carried out in an argon (Ar) atmosphere, and there was no capping layer on the alloy films in this experiment.

Rapid thermal annealing (RTA) was applied to anneal the CuMn/SiO_2/Si and CuMn/Ta/SiO_2/Si for 30 minutes. The annealing temperature was chosen between 100 and 600 °C in a N_2 atmosphere. The thickness and cross-sectional images of the films were examined with JEOL JEM-2010 TEM, while the surface morphology was measured using a Hitachi S-4800 SEM. The sheet resistance was measured with a 280SI four-point probe, and the concentration profile along the thickness direction was measured by SIMS.

III. RESULTS AND DISCUSSION

The CuMn/SiO_2 samples had been used to investigate the barrier properties in our previous study [14]. After annealing for 30 minutes, the grain grew larger and pin holes appeared at the grain boundaries, and the amount and size of these pin holes became larger with higher annealing temperature [15]. The agglomeration and grain boundaries of Cu films were very clear after annealing at 600 °C for 30 minutes. Perng *et al.* [16] and our group [14] had both mentioned that impurities in the films would affect the grain growth behavior. When the Mn concentration in the films increased to 1% and 5%, there were fewer and smaller pin holes on the surface, and grain growth was observed after annealing at 500 °C. Furthermore, the films remained continuous and smooth even after heat treatment at 600 °C. However, defects were found in the Cu- 10 at.% Mn film after 600 °C heat treatment, where the grain growth was obvious and the substrate was no longer well covered by CuMn [17]. This phenomenon reveals that both agglomeration and diffusion occurred and was caused by the decreasing in melting point of CuMn alloy where the melting point of Mn is lower than that of Cu. Judging from the aforementioned, the concentration of Mn in CuMn alloy as barrier layers should be carefully controlled.

After annealing, the grains in the Cu and Cu- 1 at.% Mn films are brick-like and the interface between the film and dielectric layer became rough after annealing at 500 °C for 30 minutes. The brick grains suggest that the grains grew and agglomerated during the annealing process. Although the grain growth behavior reduced the resistance, the pin holes were produced at the same time. In these two films, the metal films became thicker than 150 nm after annealing, so we assume that the Cu and Mn atoms both diffused into the dielectric layer. Although the self-forming layer grew between the film and dielectric layer, it could not immediately prevent the diffusion of Cu and Mn atoms and thus the diffusion occurred prior to complete formation of the self-forming layer. In the case of the Cu- 5 at.% Mn film after annealing at 500 °C for 30 minutes, no Cu atom diffusion was observed, and the resistance kept decreasing as the annealing temperature raised from 25 to 600 °C. This is because the Mn atoms continuously diffused into the interface and the Cu grains also grew up at the same time. In the case of the Cu-10 at.% Mn film after annealing at 500 °C for 30 minutes, agglomeration of Mn atoms at the interface could be observed and the Cu atoms appeared to be darker than the Mn atoms in TEM images. This is because when the Mn content increases, the grain size of Cu decreases, and thus the grain boundaries increase. This makes diffusion paths for Cu atoms to diffuse with groups of Mn atoms, so we consider that the Cu atoms will diffuse into the dielectric layer with excess Mn atoms.

In order to improve the thermal stability, a thin Ta layer was added between CnMn and SiO_2 to form a CuMn/Ta/SiO_2 stack. In this case, the main purpose is to clarify the diffusion mechanism of Mn in CuMn/Ta/SiO_2 stacks despite the thickness of metal will be increased. In Fig. 1, the resistance of each sample was measured with increasing annealing temperature. One can observe that the resistance of all samples decreased with increasing annealing temperature. During the process of rising temperature, the Mn atoms in CuMn films started diffusing toward the interface of CnMn/Ta, and thus the Mn concentration in CuMn decreased [14]. The decrease in resistance is due to the transformation of the metal film from alloy (CuMn) to pure metal (Cu). In this case, the Cu grains grew larger and grain boundaries decreased with increasing temperature. In our previous study where CuMn/SiO_2 structure without the Ta layer is used, the resistance of the Cu and Cu-10 at.% Mn films is too large to be measured after annealing at 600 °C for 30 minutes. However, in this study with the Ta layer added between CnMn and SiO_2, the resistance of each sample could be measured even after heat treatment at 600 °C, which means that the thermal stability of CuMn films on Ta/SiO_2 is better than that on SiO_2.

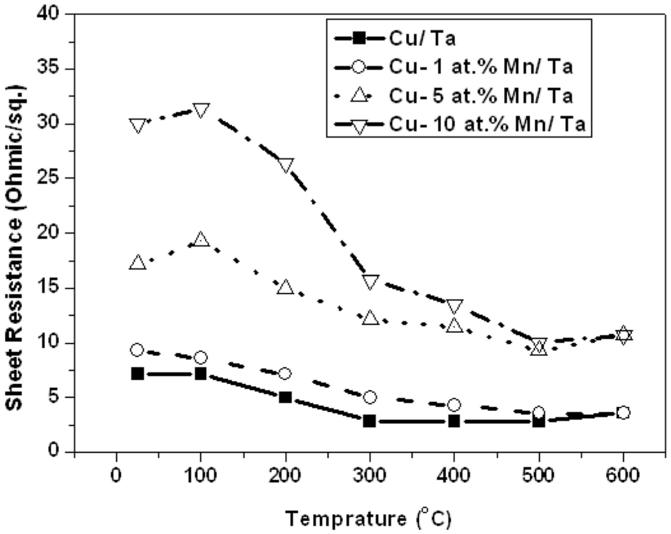

FIGURE 1. Sheet resistances of the CuMn/Ta/SiO_2 samples after annealing from 100 to 600 °C.

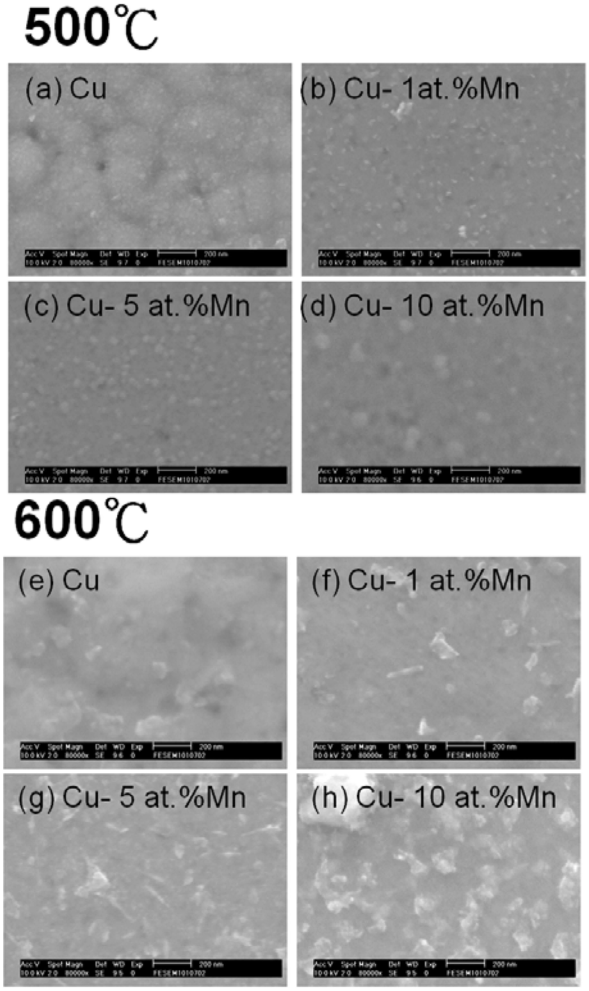

FIGURE 2. SEM images of the CuMn/Ta after RTA process with different Mn concentration. (a) Cu. (b) Cu- 1 at.% Mn. (c) Cu- 5 at.% Mn. (d) Cu- 10 at.% Mn. (e) Cu. (f) Cu- 1 at.% Mn. (g) Cu- 5 at.% Mn. (h) Cu- 10 at.% Mn.

Fig. 2 shows the top-view images of the CuMn films after heat treatment at 500 °C and 600 °C. The agglomeration of Mn on the surface increase with increasing Mn content and annealing temperature, and the surface of CuMn on Ta/SiO_2 are smoother than that on SiO_2 when the same Mn concentration is used. Smoother surface could be related to better reliability of the barrier layers. Based on the principle that atoms will diffuse along the chemical potential gradient, Mn atoms will diffuse upward to the surface of CuMn layer or downward to the interface of CuMn/Ta. Judging from the aforementioned, we can make an assumption that when a Ta layer is added, the Mn atoms will diffuse not only to the interface between CuMn and Ta but also to the grain boundaries in the Ta layer and even further to the interface between Ta and SiO_2. This assumption could be supported by the surface energy that the surface energy of Ta and SiO_2 were 2.9 J/ m^2 and 61 mJ/ m^2, respectively. The surface energy represents the ability of substrate species to be bonded to other atoms. The agglomeration of CuMn was suppressed by the added Ta layer, and the higher surface energy also improved the reliability (reduced EM and SM) of metal film stack.

Fig. 3 reveals the diffusion and self-forming behavior of barriers with the added Ta layer. All samples were treated by RTA at 500 °C for 30 minutes. In Fig. 3(a), some vacancies can be observed at the interface between Cu and Ta, which may be caused by stress releasing or the grain growth of Cu and Ta. This indicated that the metal film have poor reliability under thermal treatment. The vacancies will reduce the effective area of the metal line and cause some defect after chemical mechanical polishing (CMP). Many literatures had mentioned that pure Ta could not prevent the diffusion of Cu atoms [18]–[20]. Fig. 3(a) also shows the diffusion of Cu atoms into SiO_2. In Fig. 3(b), the diffusion of Cu is also observed in the Cu- 1 at.% Mn film, thus it would not be considered as a good barrier layer. When the Mn concentration rose to 5%, there is no diffusion of Cu into the dielectric as shown in Fig. 3(c). The continuous interface between CuMn and Ta after annealing also indicate that CuMn alloy has higher reliability on Ta than on SiO_2. By energy dispersive spectrometer (EDS), Mn atoms were detected in the Ta layer, the interface of CuMn/Ta and Ta/SiO_2. This result provides a further evidence for the assumption we made earlier. The Mn atoms diffuse to not only the interface between CuMn/Ta but also to the Ta layer and interface between Ta and SiO_2. When the Mn atoms diffuse through the diffusion paths in the Ta layer to the interface between Ta/SiO_2, a thin $MnSi_xO_y$ layer will form to be a barrier layer. When the Mn atoms diffuse into the grain boundaries of Ta layers, the diffuse paths will be blocked by the Mn atoms, and the Ta layer will then prevent the diffusion of Cu atoms. The chemical potential and the activity coefficient of Mn are shown as follows [12]:

$$\mu_{Mn} = \mu^0_{Cu} + RTLnN_{Mn} + RT\ln\gamma_{Mn} \tag{1}$$

where μ^0_{Cu} is the chemical potential of pure Cu, N_{Mn} is the atomic concentration of Mn, and RT has its usual meaning.

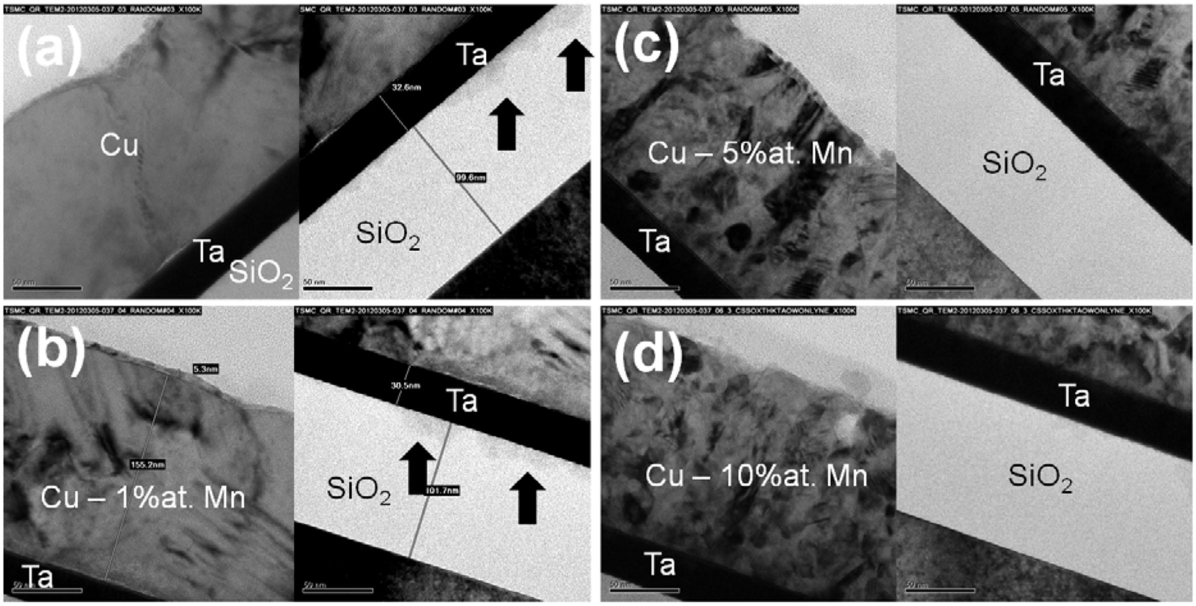

FIGURE 3. **Cross-sectional TEM images of (a) Cu/Ta. (b) Cu- 1 at.% Mn/Ta. (c) Cu- 5 at.% Mn/Ta. (d) Cu- 10 at.% Mn/Ta samples after 500 °C RTA process.**

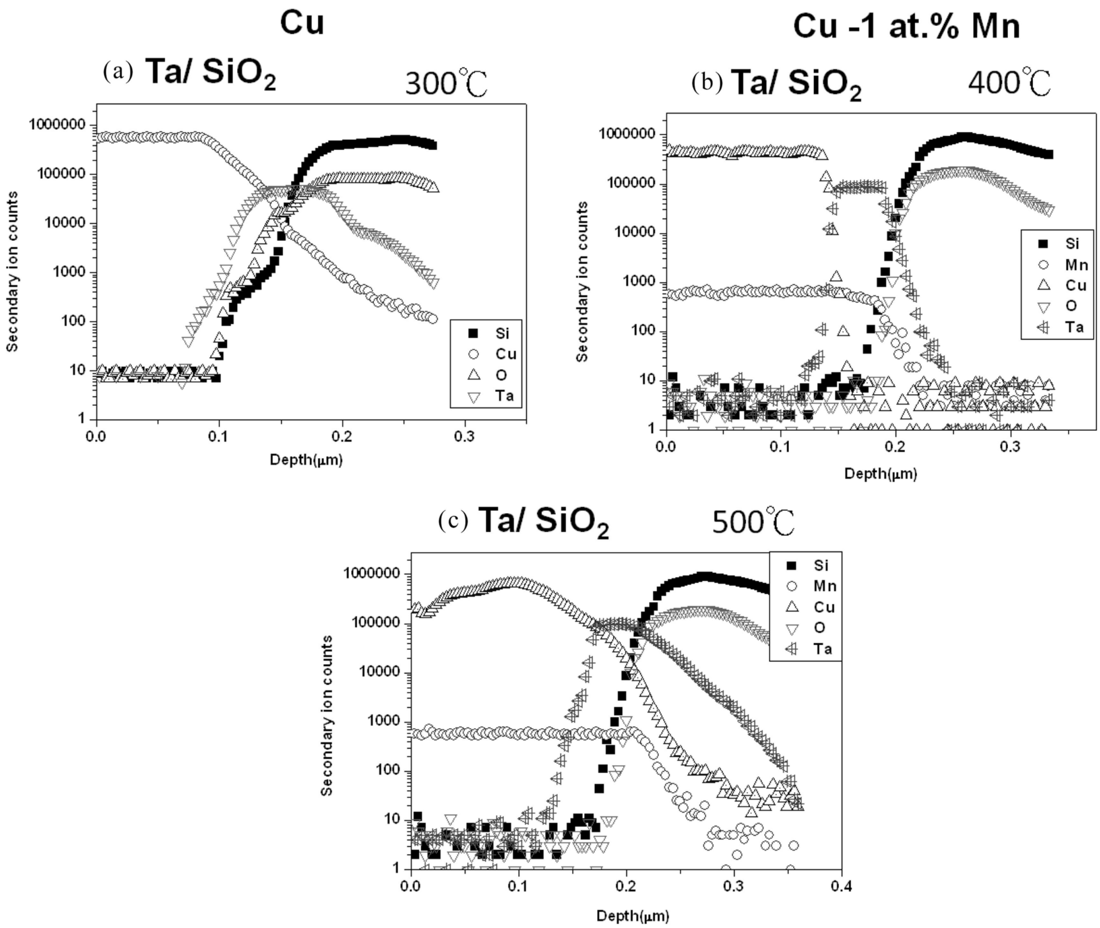

FIGURE 4. **SIMS compositional depth profiles of the CuMn/Ta after RTA process. (a) Cu/Ta/SiO$_2$. (b) and (c) Cu- 1 at.% Mn/Ta/SiO$_2$.**

In an ideal solution without any chemical interaction between Cu and Mn, γ_{Mn} is equal to 1. Because of lower chemical potential and less amount of Mn atoms in Cu- 1 at.% Mn than in Cu- 5 at.% Mn, the Mn atoms were not able to block the grain boundaries in Ta layer before the diffusion of Cu atoms. This is the reason why the Cu- 1 at.% Mn failed after

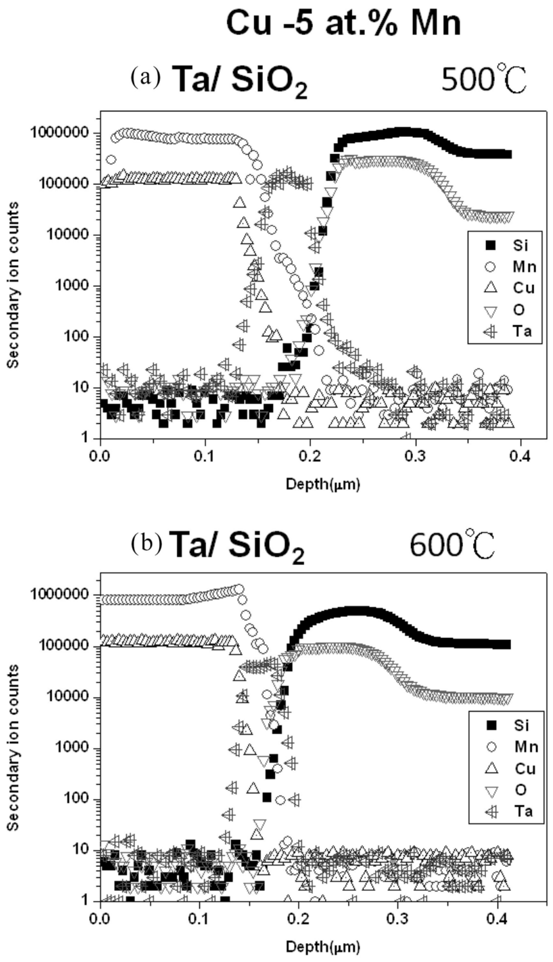

FIGURE 5. **SIMS compositional depth profiles of the Cu- 5 at.% Mn/Ta after RTA process. (a) and (b) Ta/SiO2.**

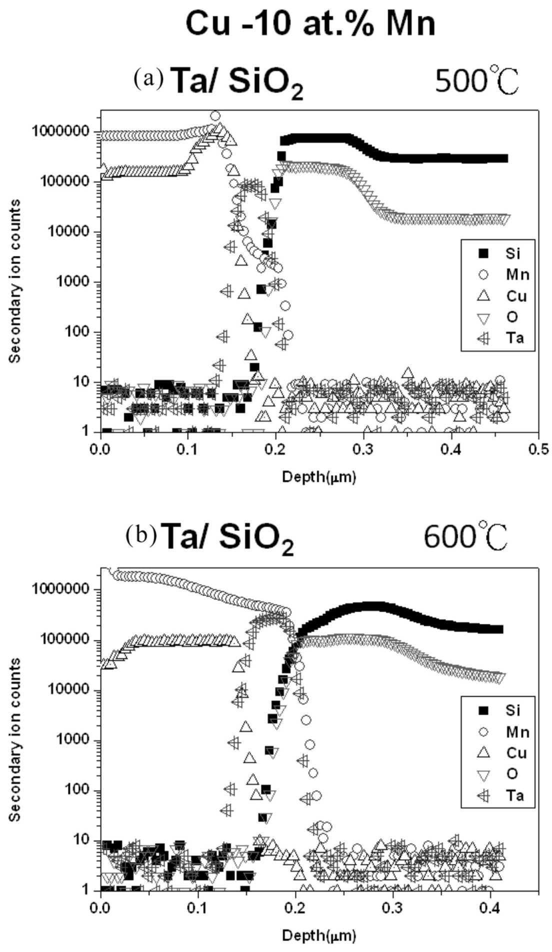

FIGURE 6. **SIMS compositional depth profiles of the Cu- 10 at.% Mn/Ta after RTA process. (a) and (b) Ta/SiO2.**

annealing at 500 °C for 30 minutes. When the Mn content rose to 10%, the chemical potential of Mn also increase, and there is no Cu observed in the dielectric as Fig. 3(d) shows. There is a difference as the Cu- 10 at.% Mn are deposited on SiO_2 and Ta/SiO_2. Our previous study has investigated the first structure of CnMn/SiO_2 where Cu atoms diffused and were covered with Mn atoms. As a result, we consider that the Cu atoms will diffuse into the dielectric layer with Mn atoms which have large chemical potential. However, in the second structure of CnMn/Ta/SiO_2, the amount of diffusion paths to the dielectric was controlled mainly by the Ta layer but not by the CuMn layer, so the window of Mn concentration in CuMn as a good barrier layer can be larger.

Figs. 4–6 show the composition distribution in the heat-treated CuMn/Ta/SiO_2. After annealing at 300 °C for 30 minutes, the Cu atoms in pure Cu films diffused into the dielectric layer as Fig. 4 shows. Fig. 4 also shows Cu- 1 at.% Mn as the barrier layer, and we can observe that the Cu signal drop off as the depth reaches the interface between CuMn and Ta layer. The self-forming layer succeeds in preventing the diffusion of Cu atoms after annealing at 400 °C for 30 minutes. However, when the annealing temperature increases to 500 °C, the Cu signal remains as the depth reaches dielectric layer. The diffusion of Cu atoms at this temperature can also be observed in the TEM image. The same phenomenon can also be observed in our previous study where no Ta layer was added [14]. As the Mn content rose to 5% and 10%, the thermal stability was enhanced. Cu atoms did not diffuse into the dielectric as Figs. 5 and 6 show. In the case of Cu- 10 at.% Mn, we don't observe Cu in the dielectric, which indicates that CuMn/Ta/SiO_2 have better barrier properties than CuMn/SiO_2.

Since the middle of 1990's, some researchers have observed solid-state amorphization (SSA) at the Cu/Ta interface when annealing at 400–600 °C [21]–[23]. This phenomenon reduces the thermal stability of Ta layer and increases the resistance of metal line. In Fig. 3(a), the interface between pure Cu and Ta layer is rough. However, in Fig. 3(b)–(d), there are a lot of Mn atoms on the surface of Ta layers and the interface is smooth. Thus we conclude that the Mn content can enhance the thermal stability of the interface between Cu and Ta. As there is enough Mn content, the barrier property of Ta can also be enhanced.

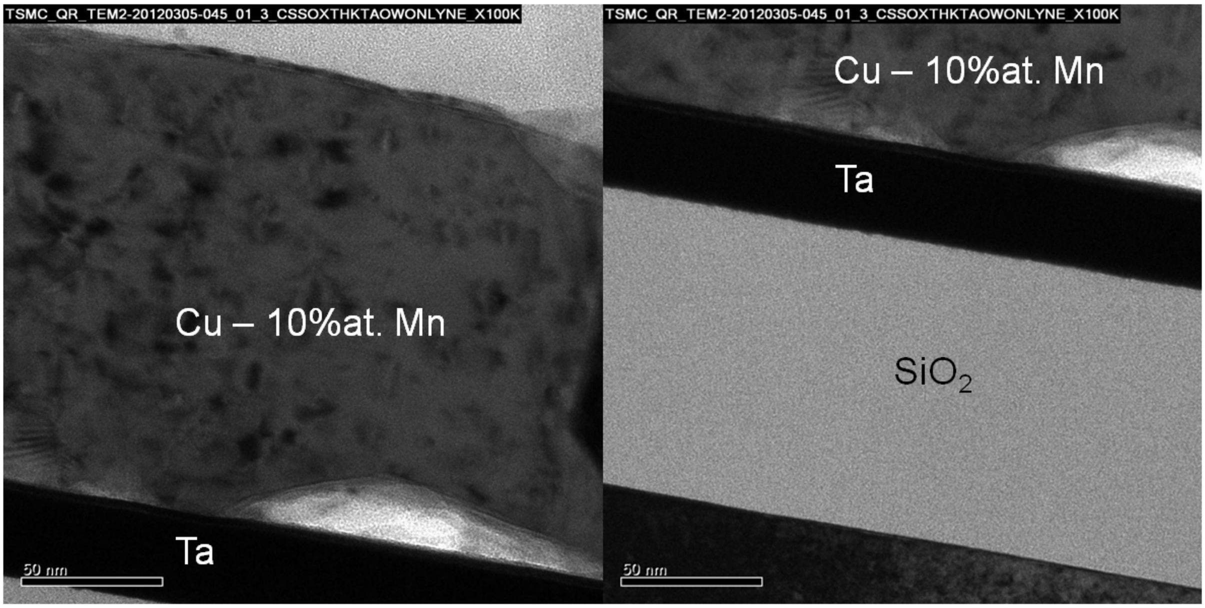

FIGURE 7. **Cross-sectional TEM images of the Cu/Cu- 10 at.% Mn/Ta samples and after 500 °C RTA process.**

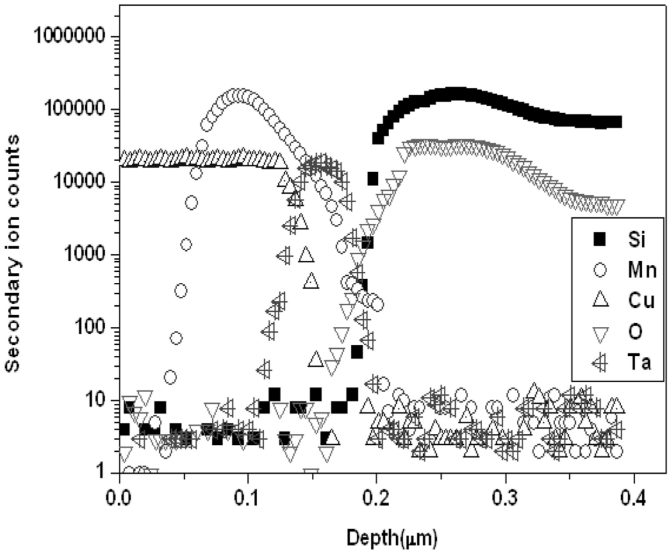

FIGURE 8. **SIMS compositional depth profiles of the Cu/Cu- 10 at.% Mn/Ta/SiO$_2$ after 500 °C RTA process.**

However, the Mn content will also increase the resistance of the barrier, and this become disadvantageous to the sequential Cu electrodeposition process. Since decreasing the Mn concentration will be detrimental to the thermal stability and barrier property, the thickness of CuMn should be reduced in replacement to maintain the resistance.

Based on the aforementioned, the thickness of CuMn was reduced to 50 nm, and the Cu- 10 at.% Mn was used to ensure enough Mn content. The sample was covered with a 100-nm-thick Cu layer and then treated by RTA at 500 °C. The TEM images are shown in Fig. 7. More Mn atoms diffused to the interface between CuMn and Ta, and no Mn atoms diffused to the surface of CuMn. One can note that when the sample are not capped with a Cu layer, the Mn atoms diffuse to the surface as shown in Fig. 3. It can be explained by the fact that pure Cu has higher chemical potential than the interface between CuMn and Ta, so the Mn atoms will tend to move toward Ta. Since the material property of Mn element is brittle in nature, it seems that the excess Mn segregation lead the film to become rough, resulting in the failure as a barrier layer. According to the result, this structure is able to make the Mn atoms diffuse toward the Ta layers and reduce the amount of Mn atoms in CuMn after heat treatment and also improves the reliability at the same time. Both the Cu- 10 at.% Mn layer and the Cu layer have high chemical potential which triggered the Mn atoms to move toward the Ta layer. Fig. 8 shows the composition distribution in the heat-treated Cu/CuMn/Ta/SiO$_2$ stack. Cu signal drop off as the depth reaches Ta layer and the Cu atoms did not diffuse into the dielectric. Moreover, the Mn atoms do not diffuse to the upper Cu layer as well.

IV. CONCLUSION

CuMn is a sensitive material when it is used as a self-forming barrier layer. In this study, we found that when a thin Ta layer is added at the interface of CuMn and SiO$_2$, the reliability and barrier properties of the CuMn barrier layer can be dramatically enhanced. In this case, the diffusion paths are controlled by the under Ta layer but not by the CuMn layer itself. The Mn atoms are found to diffuse to the grain boundaries of Ta layer and then block the diffusion paths. As a result, there will be no strict limitation for the concentration of Mn in CuMn. Further, a Ta layer with larger surface energy improves the thermal stability of CuMn layer. Neither agglomerations on the surface nor vacancies at the interface of CuMn/Ta can be found after thermal treatment. Finally, to reduce the metal resistance, the thickness of CuMn decreased from 150 nm to 50 nm and then covered with a 100-nm-thick Cu layer. The amount of Mn atoms which diffused to the Ta layer increased even when the amount of Mn atoms in the whole films decreased. Both the Cu- 10 at.% Mn and the Cu layer have high chemical potential which triggered the Mn atoms to move toward the Ta layer and reduced the amount of Mn atoms in Cu after heat treatment. In conclusion, the Cu/CuMn/Ta/SiO_2 structure could not only reduce the resistance of metal lines but also enhance the thermal stability and barrier properties.

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