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A Bilayer Diffusion Barrier of ALD-Ru/ALD-TaCN for Direct Plating of Cu

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Diffusion barrier performances of atomic layer deposited (ALD)-Ru thin films between Cu and Si were improved with the use of an underlying 2 nm thick ALD-TaCN interlayer as diffusion barrier for the direct plating of Cu. Ru was deposited by a sequential supply of *bis*(ethylcyclopentadienyl)ruthenium $[\mathrm{Ru(EtCp)_2}]$ and NH_3 plasma and TaCN by a sequential supply of $(NEt_2)_3Ta = Nbu^t$ (*tert*-butylimido-trisdiethylamido-tantalum), and H_2 plasma. Sheet resistance measurements, X-ray diffractometry, and Auger electron spectroscopy analysis showed that the bilayer diffusion barriers of ALD-Ru (12 nm)/ALD-TaCN (2 nm) and ALD-Ru (4 nm)/ALD-TaCN (2 nm) prevented the Cu diffusion up to annealing temperatures of 600 and 550°C for 30 min, respectively. This is because of the excellent diffusion barrier performance of the ALD-TaCN film against the Cu, due to its amorphous structure. A 5 nm thick ALD-TaCN film was even stable up to annealing at 650°C between Cu and Si. Transmission electron microscopy investigation, combined with energy-dispersive spectroscopy analysis, revealed that the ALD-Ru/ALD-TaCN diffusion barrier failed by the Cu diffusion through the bilayer into the Si substrate. This is due to the ALD-TaCN interlayer preventing the interfacial reaction between the Ru and Si.

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As semiconductor devices are scaled down for better performance and more functionality, the Cu-based interconnects suffer from the increase of the resistivity of the Cu wires. The resistivity increase, which is attributed to the electron scattering from grain boundaries and interfaces, needs to be addressed to further scale down semiconductor devices.[1-3] The increase in the resistivity of the interconnect can be alleviated by increasing the grain size of electroplating (EP) Cu or by modifying the Cu surface.[1] Another possible solution is to maximize the portion of the EP Cu volume in the vias or damascene structures with the conformal diffusion barrier and seed layer by optimizing their deposition processes during Cu interconnect fabrication, which are currently ionized physical vapor deposition (IPVD)-based Ta/TaN bilayer and IPVD-Cu,[4] respectively. The use of in situ etching, during IPVD of the barrier or the seed layer, has been effective in enlarging the trench volume where the Cu is filled, resulting in improved reliability and performance of the Cu-based interconnect.[5] However, the application of IPVD technology is expected to be limited eventually because of poor sidewall step coverage and the narrow top part of the damascene structures.

Recently, Ru has been suggested as a diffusion barrier that is compatible with the direct plating of Cu.[6-9] A single-layer diffusion barrier for the direct plating of Cu is desirable to optimize the resistance of the Cu interconnects because it eliminates the Cu-seed layer. However, previous studies have shown that the Ru by itself is not a suitable diffusion barrier for Cu metallization.[8,10,11] It has been reported that a physical vapor deposition (PVD) Ru film (between Cu and Si) with a thickness of 20 nm could prevent Cu diffusion up to 450°C,[8] but that a similar Ru film with a thickness of 5 nm lost its barrier property at only just above 300°C.[10] Thus, the diffusion barrier performance of the Ru film should be improved for it to be successfully incorporated as a seed layer/barrier layer for the direct plating of Cu. The improvement of its barrier performance, by modifying the Ru microstructure from columnar to amorphous (by incorporating the N into Ru during PVD), has been previously reported.[12] Another approach for improving the barrier performance of the Ru film is to use Ru just as a seed layer and combine it with superior materials to function as a diffusion barrier against the Cu. A Ru/TaN bilayer prepared by PVD has recently been suggested as a seed layer/diffusion barrier for Cu. This bilayer was stable between the Cu and Si after annealing at 700°C for 1 min.[13] The recent results on the application of Ru with respect to Cu metallization are summarized in Table I. Although these reports dealt with the possible applications of Ru for Cu metallization, cases where the Ru film was prepared by atomic layer deposition (ALD) have not been identified. These are important because of ALD's excellent conformality.

In this study, a bilayer diffusion barrier of Ru/TaCN prepared by ALD was investigated. As the addition of the third element into the transition metal nitride disrupts the crystal lattice and leads to the formation of a stable ternary amorphous material, as indicated by Nicolet,[14] ALD-TaCN is expected to improve the diffusion barrier performance of the ALD-Ru against Cu. However, the thickness of the ALD-TaCN layer should be as thin as possible to minimize the effect on the total resistance of interconnect structure, as the typical resistivity of an ALD-TaCN layer ($\sim 350\ \mu\Omega$ cm) is an order of magnitude higher than that of ALD-Ru ($\sim 12\ \mu\Omega$ cm). In this work, it is demonstrated that a bilayer diffusion barrier of ALD-Ru (4 nm)/ALD-TaCN (2 nm) prevented the Cu diffusion into Si until annealing at 550°C for 30 min. In addition, a corresponding failure mechanism of the bilayer diffusion barrier between Cu and Si was discussed, based on transmission electron microscopy (TEM) analysis. This study is of practical importance because Cu interconnect technology demands the conformal deposition of a ~ 5 nm thick diffusion barrier beyond the 28 nm technology node[15] to satisfy the requirement of the Cu interconnect, while minimizing the size effect on the Cu line resistance.

Experimental

Si wafers were used as the starting substrates. First, 2 nm thick ALD-TaCN films were deposited using sequential exposures of $(NEt_2)_3Ta = Nbu^t$ (*tert*-butylimido-trisdiethylamido-tantalum) and H_2 plasma, at a susceptor temperature of 300°C and chamber pressure of 2 Torr, in a 300 mm commercial ALD reactor (Novellus System Inc. INOVA xT reactor). Subsequently, ALD–Ru films with a thickness of 4 or 12 nm were deposited on the ALD-TaCN films (without breaking the vacuum) using sequential exposures of *bis*-(ethylcyclopentadienyl)ruthenium $[\mathrm{Ru(EtCp)_2}]$ and NH_3 plasma in another ALD chamber.

To evaluate the barrier performances of the bilayer films against Cu, 70 nm thick Cu films were deposited onto the ALD-Ru/ALD-TaCN bilayers using sputtering (Novellus System Inc. INOVA reactor). These Cu/ALD-Ru/ALD-TaCN/Si samples were then annealed in a high vacuum ($< 5 \times 10^{-5}$ Torr) for 30 min, at temperatures

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Table I. Recent results on Ru seed layer/diffusion barrier for direct plating of Cu.

Topic	Ref.	Sample structures	Results
Direct plating of Cu on Ru	6	EP Cu/Ru disk	No dewetting after annealing at 600°C of Cu/Ru in N_2 atmosphere No reaction between Cu and Ru after annealing at 800°C
	7	EP-Cu/PVD-Ru/PVD-W	EP Rh or EP Pd were also possible on PVD-Ru/PVD-W (PVD-W was used as an adhesion layer on dielectric)
	8	EP-Cu/20 nm PVD-Ru	95% of Cu plating efficiency on 20 nm PVD-Ru Good adhesion of EP Cu using scribe-peel test
	9	EP-Cu/PVD-Ru	Superfilling of Cu on Ru at the trench with an aspect ratio of 4
Diffusion barrier properties of Ru	8	PVD-Cu/20 nm PVD-Ru/Si	Interfacial stability after annealing at 450°C for 10 min under vacuum (SIMS and TEM)
	10	EP-Cu/5 nm PVD-Ru/Si	Interfacial stability at 300°C for 10 min under vacuum. (Rutherford backscattering and TEM) $RuSi_x$ formation starts at annealing at 300°C
	11	PVD-Cu/10 nm PVD-Ru/SiCOH-based low-*k* dielectric	Both Cu and Ru diffusion into the low-*k* dielectric after annealing at 300°C for 1 h under vacuum
Improvement of diffusion barrier properties of Ru	12	PVD-Cu/PVD Ru-N (10 nm)/SiO_2	Delayed silicide reaction using Ru–N compared to Ru Improved barrier performance of Ru–N between Cu and SiO_2 due to amorphous-like microstructure of Ru–N
	13	PVD-Cu/PVD-Ru (7.6 nm)/PVD-TaN (6 nm)/Si	Improved thermal stability of ion-beam deposited Ru/TaN bilayer (1 min) up to 750°C Cu_3Si formation after annealing at 500°C for 30 min in the case of Cu/Ru (10 nm)/Si structure
Effect of Ru on the properties of Cu	11	PVD-Cu/PVD-Ru/low-*k* (SiCOH-based)	Stronger Cu(111) texture on Ru compared to Ta

ranging from 450 to 700°C in increments of 50°C. After annealing, the barrier performances were evaluated using sheet resistance measurements, X-ray diffractometry (XRD) analysis, and Auger electron spectroscopy (AES) depth profiling. Cross-sectional view transmission electron microscopy (XTEM, JEOL JEM-3000F with a point-to-point resolution of 0.17 nm, equipped with a field emission gun) and energy-dispersive spectroscopy (EDS) were also performed to characterize the failure mechanism of the Ru/TaCN diffusion barrier between the Cu and Si after annealing. The image processing, including Fourier transformation of the high-resolution TEM (HRTEM) image, was conducted with Gatan image processing software called Digital Micrograph. TEM was also used for the analysis of the microstructure of the ALD-Ru and ALD-TaCN films. Plan-view bright-field (BF) TEM micrographs were digitally taken using a 1 × 1 K (1024 × 1024 pixel) multiscan charge-coupled device camera (Gatan MSC-794). Finally, for comparison, the multilayer structures of Cu/ALD-TaCN (5 nm)/Si were annealed to evaluate the diffusion barrier performance of just ALD-TaCN against Cu diffusion.

Results and Discussion

Figure 1a shows the changes in the sheet resistance of the Cu/ALD-Ru (12 nm)/ALD-TaCN (2 nm)/Si samples as a function of annealing temperature. Sheet resistance mainly represents the conditions and quantities of the Cu layer because it carries nearly all the sensor current. The sheet resistance contributions from Ru and TaCN can be neglected because of their high resistivities (Ru: 12 $\mu\Omega$ cm and TaCN: ~ 350 $\mu\Omega$ cm) and thin thicknesses. The sheet resistance of the multilayer sample does not increase with respect to that of the as-deposited sample until annealing at 550°C. Upon annealing below 500°C, the sheet resistance slightly decreases, probably due to the increase of the grain size of the Cu films. The sheet resistance starts to increase slightly after annealing at 600°C, but it increases abruptly after 650°C. To investigate the possible reasons for the increase in the sheet resistance of the multilayer structure, we performed an XRD analysis on the annealed samples.

Figure 1b shows the corresponding XRD results for the annealed samples. In the case of the as-deposited Cu/ALD-Ru (12 nm)/ALD-TaCN (2 nm)/Si sample, peaks from face-centered-cubic Cu, hexagonal-close-packed (hcp) Ru, and the Si substrate are detected. The thin-film thickness of the ALD-TaCN meant that its phases could not be identified from the XRD analysis. Figure 1b clearly shows that the initial multilayer structure of Cu/ALD-Ru (12 nm)/ALD-TaCN (2 nm)/Si is preserved until an annealing temperature of 600°C. It also shows that Cu silicides are first detected after annealing at 650°C, when the sudden increase of sheet resistance was observed. This indicates that the drastic increase in sheet resistance, discussed in the previous paragraph, is mainly caused by the consumption of Cu and the formation of Cu silicide via the diffusion of Cu into Si through the Ru/TaCN bilayer, which has a much higher resistivity than Cu.[16] The dominant Cu silicide phase formed is η''-Cu_3Si, which is consistent with the results obtained from an annealing study of Cu/single-layer ALD diffusion barrier/Si structures.[17,18]

The compositional profile across the bilayer diffusion barrier was investigated using AES depth profiling (Fig. 2). As is expected from the XRD results, no significant intermixing occurs, even after annealing at 600°C (Fig. 2b). However, some change might be shown in the composition profiles of Cu and Ru compared to those of the as-deposited sample (Fig. 2a). The widening of the Cu and Ru profiles after annealing at 600°C is not considered to be due to the intermixing between layers. Rather, we think that the roughening of the annealed Cu or Ru film surface (caused by grain growth during heat-treatment) makes the profiles of the annealed samples broader than those of the as-deposited sample. From Fig. 2c, we can confirm that Cu diffuses into the Si substrate, and Si diffuses out to the Cu after annealing at 650°C, indicating the failure of the diffusion barrier.

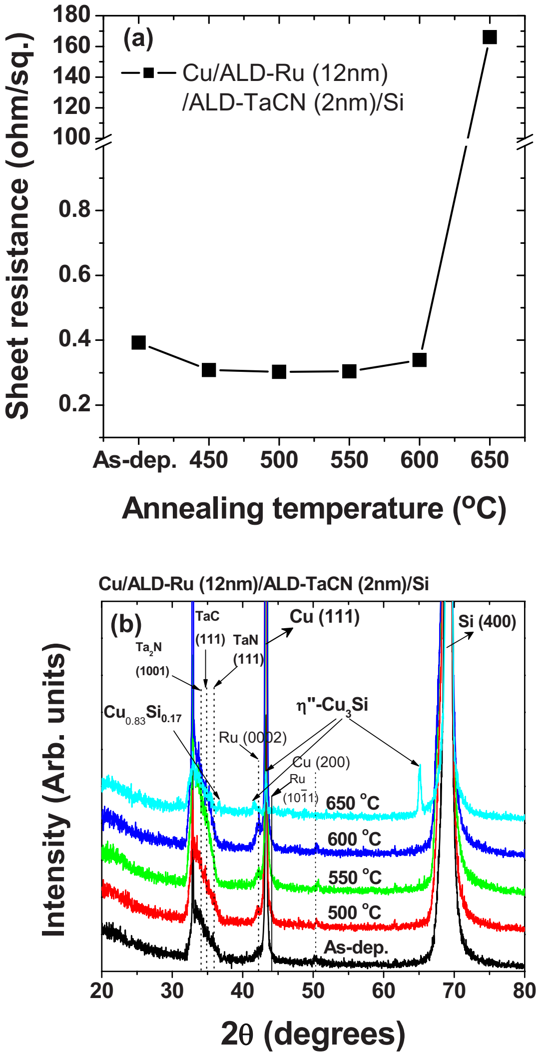

Figure 1. (Color online) (a) Sheet resistance changes and (b) XRD patterns of the Cu/ALD-Ru (12 nm)/ALD-TaCN (2 nm)/Si samples as a function of annealing temperature.

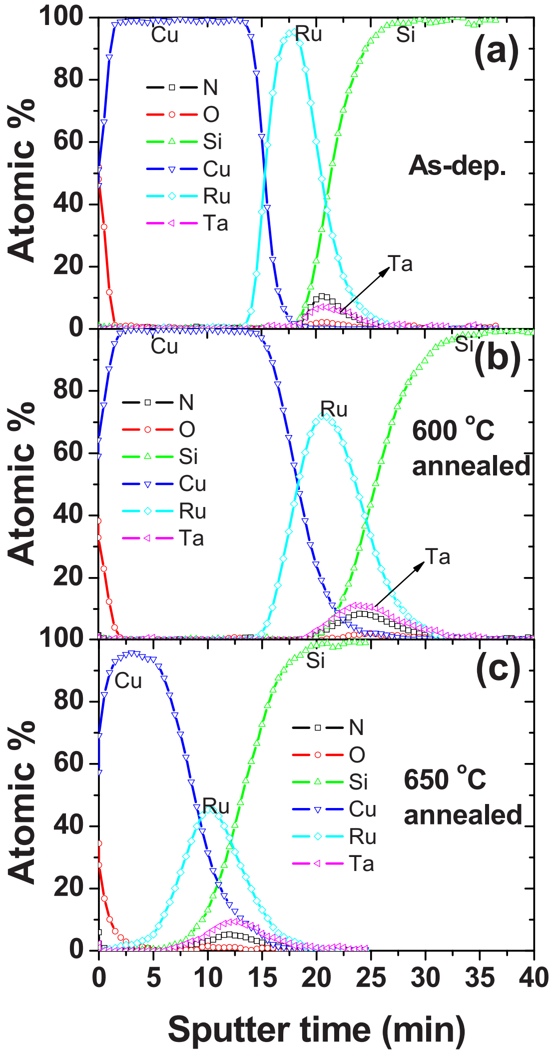

Figure 2. (Color online) AES depth profiles of the Cu/ALD-Ru (12 nm)/ALD-TaCN (2 nm)/Si samples: (a) as-deposited, (b) annealed at 600°C for 30 min, and (c) annealed at 650°C for 30 min.

To thoroughly examine the possible interfacial reaction, the interface morphology after annealing, and the failure mechanism of the bilayer diffusion barrier, XTEM analyses were performed for annealed samples. Figure 3 shows a high-resolution XTEM image of the Cu/ALD-Ru (12 nm)/ALD-TaCN (2 nm)/Si sample after annealing at 600°C for 30 min. The XTEM image does not show the formation of Cu silicides or any interfacial reactions between the Ru and the TaCN or the TaCN and the Si with flat interfaces between layers. Fourier transformation of the HRTEM image in the Ru region shows that the ALD-Ru film preserves its original hcp phase (the inset of Fig. 3), but the grain size and roughness of the Ru is increased compared to that of the as-deposited sample (not shown here). This is consistent with the XRD results, which show the peak arising from the Ru(0002) plane to increase in intensity as the annealing temperature is increased to 600°C (Fig. 1b).

However, the XTEM image of the sample annealed at 650°C (Fig. 4a) shows a drastic change in the multilayer structure. A new phase, triangular in shape, with a size of 2–3 μm was formed. This was identified as a slightly Si-deficient η″-Cu_3Si phase from the use of EDS analysis and electron diffraction. Also, the rupture of the Ru layer is observed from TEM. This is due to a large volume expansion during the formation of copper silicide (the unit cell volume of Cu_3Si is 46 Å^3, while that of Si is 20 Å^3).[19] However, as can be seen from the image of the same sample obtained at a different location (Fig. 4b), the Cu/Ru/TaCN/Si structure is still present. In addition, even at the location where the failure occurred (Fig. 4a), Ru still preserves its layer. Generally, the failure of a diffusion barrier between Cu and Si occurs by two different mechanisms.[20,21]

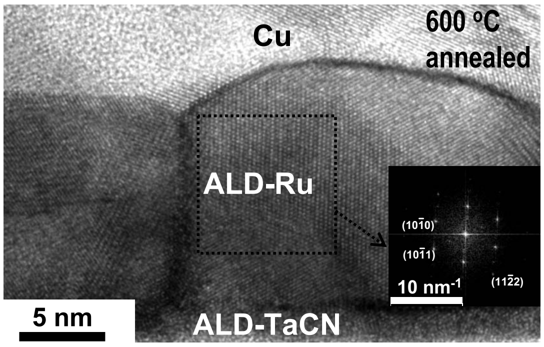

Figure 3. Cross-sectional view HRTEM images of the Cu/ALD-Ru (12 nm)/ALD-TaCN (2 nm)/Si samples annealed at 600°C for 30 min. The inset shows the Fourier transformation of the HRTEM image of the region where the ALD-Ru layer is initially located.

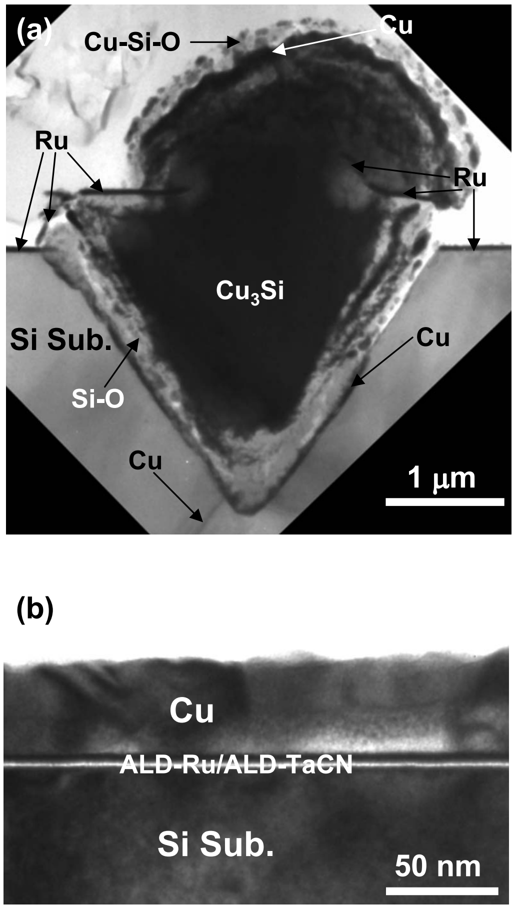

Figure 4. Cross-sectional view BF TEM images of the ALD-Ru (12 nm)/ALD-TaCN (2 nm)/Si samples annealed at 650°C for 30 min. The images were taken at different locations on the same TEM specimen: (a) TEM image taken from the intact diffusion barrier and (b) TEM image taken from the failed diffusion barrier with the formation of η''-Cu_3Si.

One is the diffusion of Cu through the barrier layer, resulting in the formation of crystalline defects in the Si or copper silicides. The other mechanism involves interfacial reactions between the barrier layer and Si, which can be best depicted by using a phase diagram. It has been reported that the failure of the Ru diffusion barrier between Cu and Si started by the formation of Ru silicide at 300°C.[10] As the annealing temperature increased, the Ru layer was completely converted to Ru silicide and eventually failed by the diffusion of Cu through the Ru silicide into the Si. This indicates that a Ru film functions as a sacrificial barrier between Cu and Si.[20] In this study, by inserting an ultrathin layer of TaCN between the Ru and Si, the reaction between the Ru and Si was prevented. Ternary phase diagrams of Ta–C–Si and Ta–N–Si at 700°C [22] have shown that both tantalum nitrides and carbides are thermodynamically unstable with respect to Si. Laurila et al.[23] investigated the failure mechanism of a TaC diffusion barrier between Cu and Si. They found that the initial failure of the diffusion barrier occurred by Cu diffusion into Si, forming Cu_3Si, without any interfacial reaction at the annealing temperature of 750°C. When the annealing temperature was increased to 800°C, the interfacial reaction between TaC and Si eventually took place, resulting in the formation of SiC and $TaSi_2$. Min et al.[21] also showed that the predominant failure mechanism of TaN, when between Cu and Si, was the diffusion of Cu into Si, not the interfacial reactions. Our results indicate that the interface between TaCN and Si is quite stable even up to 650°C, similar to the interfaces between TaN and Si or TaC and Si. For this reason, the barrier failure of the Ru/TaCN bilayer occurs by the diffusion of Cu through the barrier layer. This suggests that the diffusion barrier performance of the bilayer will be determined by the performance of the TaCN.

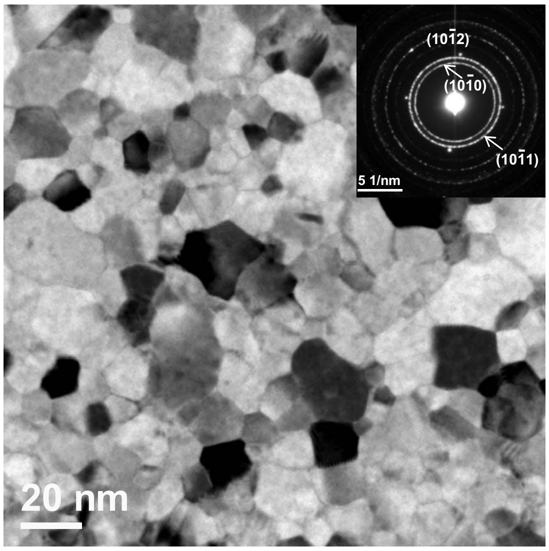

Figure 5. Plan-view TEM BF image of the ALD-Ru film. The inset is the corresponding selected-area electron diffraction pattern.

It has previously been reported that a sputter-deposited Ru film with a thickness of 20 nm prevented Cu diffusion up to 450°C, while a 5 nm thick Ru single layer failed as a barrier against Cu diffusion at temperatures only just above 300°C based on TEM and secondary ion mass spectrometry (SIMS) analyses.[8,10] We note that the sputter-deposited Ru film formed a columnar microstructure oriented vertically with respect to the Si substrate. Figure 5 shows a plan-view TEM BF micrograph and a selected area diffraction pattern (SADP) for the ALD-Ru film in this study. It shows that the ALD-Ru film forms a polycrystalline microstructure with the grain sizes in the range of approximately 10–25 nm. Between the Ru grains, the grain boundaries are clearly observed. Thus, we think that the formation of polycrystalline columnar grains of ALD-Ru is a possible reason for its poor diffusion barrier performance against Cu, because it would provide a fast diffusion pathway for Cu into Si.[24]

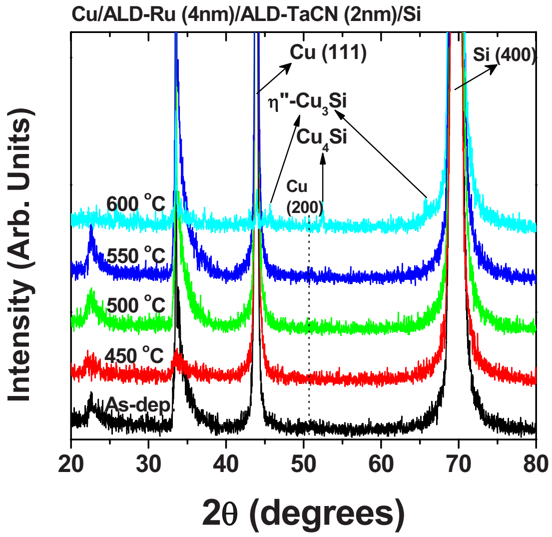

Figure 6. (Color online) XRD patterns of the Cu/ALD-Ru 4 (nm)/ALD-TaCN (2 nm)/Si samples as a function of annealing temperature.

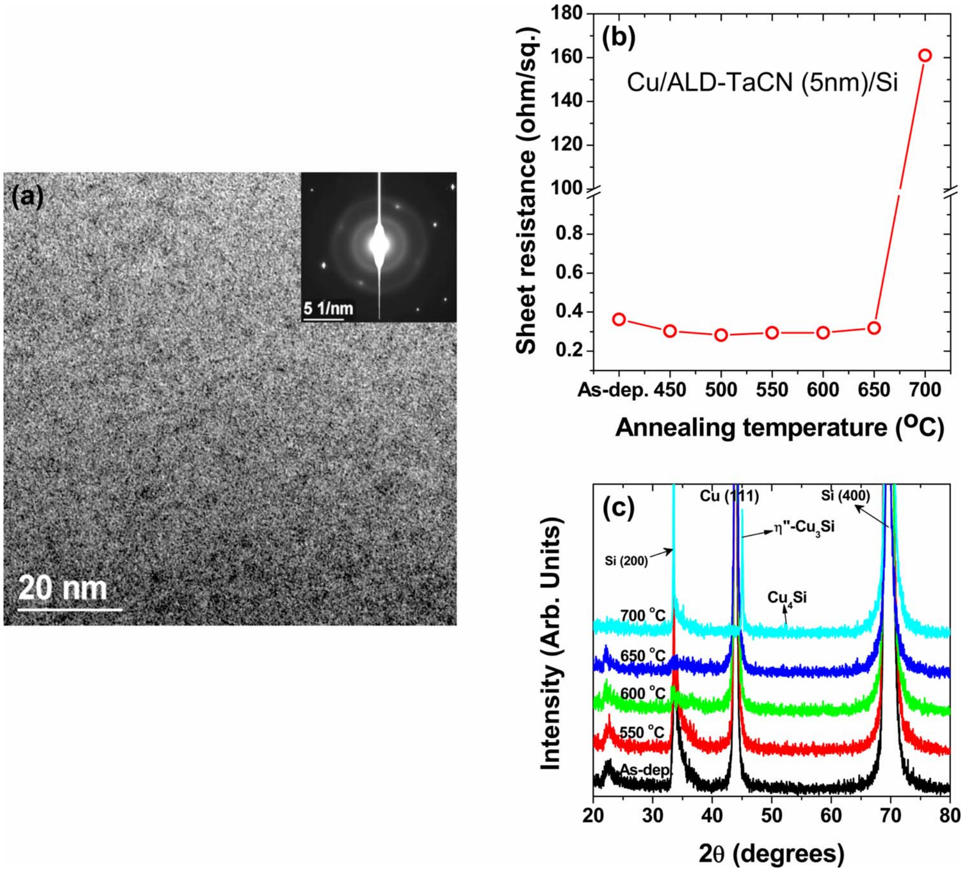

Figure 7. (Color online) (a) Plan-view TEM BF images of the ALD-TaCN film and the corresponding selected-area electron diffraction pattern. (b) Sheet resistance changes of the Cu/ALD-TaCN (5 nm)/Si samples. (c) XRD patterns of the Cu/ALD-TaCN (5 nm)/Si samples as a function of annealing temperature.

Our study clearly shows that the diffusion barrier performance of the ALD-Ru film between the Cu and Si is significantly improved by inserting a very thin (2 nm thick) ALD-TaCN film under the Ru film. The XRD analysis confirmed that the bilayer diffusion barrier with a total thickness of 14 nm was stable up to 600°C (Fig. 1b). XRD analysis for the bilayer diffusion barrier of Ru/TaCN with a total thickness of ~6 nm (Fig. 6) also showed that it was stable enough to be integrated into the present Cu wiring technology, which generally requires thermal stability up to ~400°C, due to the integration of low-*k* materials. With the bilayer with the thickness of 6 nm, the diffusion of Cu into Si through the bilayer diffusion barrier, forming Cu silicide, happened after annealing at 600°C.

To provide a possible explanation for the improved performance of the bilayer diffusion barrier upon inserting a very thin ALD-TaCN layer, we investigated the microstructure of the ALD-TaCN film. The plan-view TEM BF image of the ALD-TaCN film (Fig. 7a) showed a featureless and very uniform contrast, indicating that the film formed was either amorphous or had a nanocrystalline structure close to being amorphous. From the corresponding broad halos in the SADP shown in the inset of Fig. 7a, it is confirmed that the ALD-TaCN forms an amorphous phase. To characterize the barrier performance of a single ALD-TaCN film against Cu, we conducted the sheet resistance measurement and XRD after annealing at various temperatures (Fig. 7b). The sheet resistance of the Cu/ALD-TaCN (5 nm)/Si structure was maintained at its initial state up to an annealing temperature of 650°C, indicating that a very thin layer of ALD-TaCN can effectively prevent the Cu diffusion into Si at temperatures as high as 650°C. In fact, we could not observe any Cu silicide peaks after annealing at 650°C from the XRD analysis (Fig. 7c). In addition, an η''-Cu_3Si phase formation was observed in the XRD only after annealing at 700°C. These results indicated that the improved diffusion barrier performance of the bilayer of ALD-Ru/ALD-TaCN is due to the superior diffusion barrier performance of the ALD-TaCN film, due to it having an amorphous structure against the Cu, which means it is free of grain boundaries that could act as fast diffusion paths even at moderate temperatures.

Conclusions

We investigated the diffusion barrier of an ALD-Ru/ALD-TaCN (2 nm) bilayer using sheet resistance measurements, XRD, and TEM for forming the seed layer/diffusion barrier for direct plating of Cu. A sheet resistance measurement, XRD, and AES depth profiling showed that the bilayer diffusion barrier of ALD-Ru (12 nm)/ALD-TaCN (2 nm) was stable up to 600°C for 30 min, while that of ALD-Ru (4 nm)/ALD-TaCN (2 nm) was able to prevent the Cu diffusion up to 550°C for 30 min. A possible reason for such good barrier performances is the excellent diffusion barrier property of ALD-TaCN film, due to it having an amorphous phase. A 5 nm thick ALD-TaCN single layer was stable up to annealing at 650°C between Cu and Si. Finally, based on the XTEM analysis, combined with EDS, it was found that the bilayer diffusion barrier failed by the diffusion of Cu into Si through the bilayer, not by interfacial reactions between layers. The good diffusion barrier performance of the bilayer of ALD-Ru/ALD-TaCN for blanket films emphasizes that this design can also be successfully integrated into a Cu interconnect with a high-aspect ratio because the ALD process has the capability of conformal deposition. We think that the design of the ALD-Ru/ALD-TaCN bilayer evaluated in this study is also beneficial for reducing the resistance increase in the Cu wiring caused by the size effect. This is due to the excellent step coverage of the ALD process and the possibility of direct plating eliminating the seed layer.

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