Thermal stability of selective chemical vapor deposited tungsten contact and effects of in situ N₂ plasma treatment

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This work investigates the thermal stability of Al/W/p$^+-n$ junction diodes, in which the W contact was filled using selective chemical vapor deposition to a thickness of about 450 nm and served as diffusion barrier between the Al and the Si substrate. The effects of in situ N$_2$ plasma treatment on the barrier effectiveness were also investigated. The Al/W(450 nm)/p$^+-n$ junction diodes can sustain a 30 min furnace annealing up to 575 °C. With an in situ N$_2$ plasma treatment on the W surface caused a thin layer of WN$_x$ to form on the W surface, and the nitrified layer of WN$_x$/W acting as barrier between the Al and the Si substrate effectively suppressed WAl$_{12}$ formation at elevated temperatures, resulting in a significant barrier improvement. N$_2$ plasma treatment at 100 W for 300 s enabled the Al/WN$_x$/W(450 nm)/p$^+-n$ junction diodes to sustain thermal annealing at temperatures up to 625 °C without degradation of electrical characteristics.

I. INTRODUCTION

Because Si is highly soluble in Al alloy and exhibits high diffusivity along Al grain boundaries, Al/Si contacts tend to fail due to Si dissolving in Al and Al spiking into Si substrates. The Al–Si alloy also becomes supersaturated during cooling cycles and excess Si precipitates at the Al/Si interface, resulting in degradation of contact characteristics. Therefore, the introduction of a barrier layer between the Al metallization and the Si substrate is of fundamental importance in preventing junction spiking and avoiding contact degradation.

Tungsten has been considered as a good contact barrier for protecting shallow junctions from aluminum spiking and achieving low contact resistance. Tungsten nitride is also an effective diffusion barrier between Si and contact metals because it is chemically more stable than pure tungsten, and has a greater capacity for suppressing interdiffusion between Al and Si. Generally, tungsten nitride is deposited by reactive sputtering of a pure W target in Ar–N$_2$ mixed ambient. It has been reported that reactively sputtered tungsten nitride far surpassed pure W metal as a diffusion barrier between Al and Si substrates. However, it is difficult to deposit tungsten nitride with excellent barrier properties into contact holes of submicron dimension using sputtering techniques because of potential step coverage problems. In this respect, selective chemical vapor deposition of tungsten (selective CVD-W) is a most attractive technique for filling deep submicron contact holes in ultralarge scale integrated (ULSI) interconnect applications.

It has been reported that a selective 73-nm-thick CVD-W film acted as an effective diffusion barrier between Al and Si substrate at 450 °C for 30 min; however, it failed at 520 °C. It has also been reported that no discernible reaction took place at an Al/W interface at temperatures up to 500 °C with a W film deposited using CVD; however, reaction occurred at an Al/W interface with sputtered W even at 450 °C and a WAl$_{12}$ compound formed. Moreover, it was found that N$_2$ plasma treatment of a CVD-W surface before Al deposition prevents Al–W alloy formation and suppresses the increase of sheet resistance for the Al/W structure. Although these studies have provided much valuable information for the application of thin CVD-W films (less than 100 nm) as diffusion barriers, nevertheless, little study has been made of the thermal stability of thicker CVD-W film.

Generally, W plugs used in industry are 450 nm or more in thickness. In this study, 100 and 450-nm-thick CVD-W films were selectively deposited to fill contact holes and acted as diffusion barriers between Al and Si substrates. The film resistivities of the selective CVD-W were found to be about 10 $\mu\Omega$ cm. This technique offers the advantage of producing a fully self-aligning contacts and barrier-formation processes. In addition, the resultant structures also possess more planar surfaces for subsequent metallization processing. To study the effects of N$_2$ plasma treatment, an in situ N$_2$ plasma treatment was performed on tungsten surfaces prior to Al metallization. After the treatment, thin WN$_x$ layers formed on the surfaces of the W layers, which efficiently suppressed diffusion between W and Al during post-metallization thermal annealing. The results of this study might be useful in producing selective CVD-W-filled plugs for ULSI interconnection applications.

II. EXPERIMENT

To evaluate the barrier effectiveness of a selective CVD-W layer, various samples of Al/barrier/p$^+-n$ junction diodes were prepared. The starting material was n type, (100)-oriented Si wafers with 4–7 $\Omega$ cm nominal resistivity. After RCA standard cleaning, the wafers were thermally oxi-
dized to grow a 500 nm oxide layer. Diffusion areas with sizes of $500 \times 500$ and $1000 \times 1000 \mu m^2$ were defined on the oxide-covered wafers using conventional photolithographic techniques. The $p^+ - n$ junctions 300 nm deep were formed using BF$_2$ implantation at 30 keV to $3 \times 10^{15} \text{cm}^{-2}$ followed by furnace annealing at 900 °C for 30 min in N$_2$ ambient.

After junction formation, the wafers were split into four groups for the preparation using various metallization structures: Al/$p^+ - n$, Al/W(100 nm)/$p^+ - n$, Al/W(450 nm)/$p^+ - n$, and Al/WN$_x$/W(450 nm)/$p^+ - n$. For the Al/W(100 nm)/$p^+ - n$ and Al/W(450 nm)/$p^+ - n$ diodes, the contact holes were selectively deposited with CVD-W layers of 100 and 450 nm, respectively, followed by Al metallization. For the Al/WN$_x$/W(450 nm)/$p^+ - n$ diodes, an in situ N$_2$ plasma treatment was performed on the selectively deposited CVD-W layers of 450 nm thickness before the Al metallization; thus, thin WN$_x$ layers formed on the W surfaces. The schematic cross sections of these differently metallized $p^+ - n$ junction diodes are shown in Fig. 1. The contact holes in the Al/W(450 nm)/$p^+ - n$ and Al/WN$_x$/W(450 nm)/$p^+ - n$ diodes were almost fully filled by selective CVD-W; this provided nearly planar surfaces for subsequent metallization processing.

Prior to selective CVD-W deposition, the wafers were dipped in dilute HF (50:1) for 30 s, followed by a rinse in deionized water for 3 min and a spin dry. The wafers were then loaded into a load-locked cold wall W-CVD system (ULVAC ERA-1000S) within 5 min and transferred to the deposition chamber without exposure to the atmosphere. The ERA-1000S is a fully automatic single wafer CVD system equipped with a cluster of multichambers, including a load/unload, buffer and two deposition chambers. This system employs a robot unit in the buffer chamber for wafer transfer in vacuum. The aluminum-alloy reactor was watercooled and was kept at a high vacuum base pressure by a turbopump. The base pressure of the CVD chamber was $1 \times 10^{-6}$ Torr.

In this study, W films were chemically vapor deposited using the SiH$_4$ reduction of WF$_6$ process under the following conditions: substrate temperature 300 °C, total gas pressure 100 mTorr, WF$_6$ flow rate 40 sccm, SiH$_4$ flow rate 10 sccm, and H$_2$ carrier gas flow rate 1000 sccm. After selective CVD-W deposition, one group of wafers was further treated with in situ N$_2$ plasma without breaking the vacuum under the following conditions: N$_2$ flow rate 80 sccm, total gas pressure 30 mTorr, treatment time 300 s, and plasma generation power ranging from 50 to 200 W. Finally, Al metallization was applied to all samples.

To investigate thermal stability of the differently metallized junction diodes, samples were thermally annealed in an N$_2$ flow furnace for 30 min at temperatures ranging from 350 to 650 °C. Electrical characteristics were measured using an HP-4145B semiconductor parameter analyzer. Unpatterned samples with various W/Si, Al/W/Si, and Al/WN$_x$/W/with structures were also prepared for material analysis. Sheet resistance of the multilayer structures was measured using a four-point probe. X-ray diffraction (XRD) analysis was used for phase identification. Auger electron spectroscopy (AES) was used to observe WN$_x$ formation following the N$_2$ plasma treatment. Scanning electron microscopy (SEM) was used to observe surface morphology as well as cross-sectional microstructure, and secondary ion mass spectroscopy (SIMS) was used for elemental depth profile measurement.

III. RESULTS AND DISCUSSION

A. Selective CVD-W and N$_2$ plasma treatment

Figure 2 shows overfilled 0.5 μm contact holes resulting from using a selective CVD-W technique. Excellent selectivity, good uniformity, and low resistivity W (about 10 μΩ cm) were obtained with a WF$_6$/SiH$_4$ flow rate of 40/10 sccm. Thus, this WF$_6$/SiH$_4$ flow rate was used to de-
posit tungsten during barrier study in this work. Details of the CVD-W selectivity study were reported elsewhere 17 and are not discussed further in this article.

Post-deposition N₂ plasma treatment resulted in a sheet resistance change in the W/Si samples. Figure 3 shows the percentage change of sheet resistance versus N₂ plasma power for a W(450 nm)/Si sample. The sheet resistance increased slightly with increasing plasma power, presumably due to formation of thin WNₓ layers on W surfaces, and the thicknesses of WNₓ layers increased with N₂ plasma power. The measured AES depth profiles, as shown in Fig. 4, provide evidence for these presumptions. With N₂ plasma treatment at 100 W, a thin WNₓ layer about 2 nm thick was formed on the W surfaces [Fig. 4(b)], while WNₓ layer thicknesses were about 5 nm in the samples treated with N₂ plasma at 200 W [Fig. 4(c)]. Since metal nitride generally has a higher resistivity than corresponding pure metal, the presence of a thin WNₓ layer on the W surface obviously resulted in a slight increase in sheet resistance in the W/Si samples.

B. Sheet resistance measurement and XRD analysis

Sheet resistance measurement and XRD analysis were used to evaluate the barrier capability of the W films. Figure 5 shows the percentage change in sheet resistance plotted against annealing temperature for the W/Si, Al/W/Si, and Al/WNₓ/W/Si samples. The Al/W/Si samples whose W barriers did not receive N₂ plasma treatment showed increases in sheet resistance after annealing at 550 °C. With N₂ plasma treatment on the W surface, sheet resistance for the Al/WNₓ/W/Si samples remained constant up to at least 575 °C. Although the WNₓ layer was very thin, it showed an excellent barrier ability to suppress the reaction between Al and W, resulting in improved thermal stability for the Al/WNₓ/W/Si structure.

The increase in sheet resistance for the Al/W/Si structure reflects the consumption of conductive aluminum due to WAl₁₂ formation, as confirmed by the x-ray diffraction pattern shown in Fig. 6. The WAl₁₂ compound appeared for the Al/W/Si sample annealed at 550 °C [Fig. 6(b)]. After annealing at 600 °C, phase signal intensity of the WAl₁₂ increased and a part of it was converted into WAl₅ phase. W₅Si₃ and W₅Si₂ signals both appeared as well [Fig. 6(c)]. Moreover, the Al signal disappeared while the α-W signal remained, implying that the 500 nm Al layer had been completely converted into WAl₁₂ and WAl₅ but the 450 nm W layer was only partially consumed. The presence of W₅Si₃ and W₅Si₂ signals indicates that reactions took place at the W/Si interface during the 600 °C annealing.

The results of XRD analysis of Al/WNₓ/W/Si samples treated with N₂ plasma at 200 W are illustrated in Fig. 7. No compound phase of W–Al or W–Si was observed after samples were annealed at temperatures up to 600 °C [Fig. 7(b)], which confirms the integrity of the multilayer structure. Apparently, N₂ plasma treatment has the effect of increasing the thermal stability of the Al/barrier/Si structure. After annealing at 650 °C, W(Si, Al)₂ and WSi₂ signals ap-
peared and the Al signal remained, while no WAl12-related signals were observed [Fig. 7(c)]. The W/Si samples without Al overlayers were also investigated for comparison. The increase in sheet resistance for the W/Si structure after annealing at temperatures above 700 °C presumably resulted from the consumption of conductive tungsten due to the formation of WSi2, as confirmed by the XRD analysis results shown in Fig. 8. In fact, the XRD spectra show that weak WSi2-phase signals began appearing at 650 °C, indicating reaction between W and the Si substrate. The WSi2-signal intensity increased with increasing annealing temperature, but the α-W signal remained even after annealing at 750 °C. This indicates that the 450 nm W layer was only partially consumed after annealing at 750 °C. By comparing the XRD spectra of W/Si samples (Fig. 8) with those of Al/W/Si and Al/WNₓ/W/Si samples (Figs. 6 and 7), we found that the formation of WAl12 or W(Si, Al)2 promoted the formation of WSi2 for the Al/W/Si structure. Similar results have been reported in which the presence of an Al overlayer in the Al/W/Si structure led to a reduction in thermal stability of the underlying W/Si interface.5 Comparative thermal stability results for the various layered structures are given in Table I.

C. Electrical measurements

Barrier capability of the self-aligned selective CVD-W layers was investigated by evaluating the thermal stability of the Al/W/p⁺-n junction diodes using electrical measurements. Figure 9 illustrates the distributions of reverse bias leakage current density measured at −5 V for the Al/p⁺-n, Al/W(100 nm)/p⁺-n, and Al/W(450 nm)/p⁺-n junction diodes annealed at various temperatures; the diodes had a diffusion area of 500×500 or 1000×1000 μm², and at least 30 diodes were measured in each case. Electrical characteristics of the Al/p⁺-n diodes deteriorated after annealing at 400 °C [Fig. 9(a)], while the Al/W(100 nm)/p⁺-n diodes remained stable up to 500 °C [Fig. 9(b)]. Moreover, the Al/W(450 nm)/p⁺-n diodes were able to retain their integrity up to 575 °C [Fig. 9(c)]. It is clear that the thermal stability of the Al/W/p⁺-n diodes can be improved further by using a thicker W film. Reaction of Al and W at elevated temperatures leads to the formation of WAl12, and the barrier capability of a thin W film will be determined by the W consumption. For the Al/W(450 nm)/p⁺-n diodes, partial consumption of the thicker W layer due to the WAl12 forma-
tion did not deteriorate the integrity of W/Si interface; thus, the diodes were stable even after annealing at 575 °C.

For comparison, junction diodes with W(450 nm)/p⁺-n structures, but without Al overlayers were also fabricated for thermal stability study. Figure 10 shows that the W(450 nm)/p⁺-n diodes retained their integrity up to 700 °C and showed only slight degradation after annealing at 750 °C. This implies that degradation of the Al/W(450 nm)/p⁺-n diodes can be attributed to the presence of the Al overlayer.

The barrier capability of CVD-W layer can be effectively improved by in situ N₂ plasma treatment prior to Al metallization. Figure 11 shows the statistical distributions of reverse bias leakage current density for the variously annealed Al/WNₓ/W(450 nm)/p⁺-n junction diodes, in which the WNₓ layer on the W surface was formed by N₂ plasma treatment. About half of the diodes whose W barriers were treated with N₂ plasma at 50 W survived thermal annealing at 600 °C, retaining a leakage current density of less than 100 nA/cm², as shown in Fig. 11(a); by contrast, none of the Al/W(450 nm)/p⁺-n diodes survived the same thermal annealing at 600 °C [Fig. 9(c)]. Because the thickness of the WNₓ layer increased with increasing N₂ plasma power (Fig. 4), further improvement of barrier capability can be obtained by increasing the N₂ plasma power. With N₂ plasma treatment at 100 W, the Al/WNₓ/W(450 nm)/p⁺-n diodes were able to retain their integrity up to 625 °C, as shown in Fig. 11(b). Increasing the N₂ plasma power to 200 W did not improve the thermal stability of the Al/WNₓ/W(450 nm)/Al/WNₓ/W(450 nm)/p⁺-n diodes, as shown in Fig. 11(c). Thermal stability temperatures for Al/WNₓ/W(450 nm)/p⁺-n diodes as high as 650 °C were not achieved in this study, presumably because of closeness to the Al melting point of 660 °C, and the formation of WSi₂ at the W/Si interface (Fig. 7). Based on these experimental results, we conclude that selective CVD-W layers with post-deposition in situ N₂ plasma treatment form effective diffusion barrier between Al and Si substrate.

### Table I. Comparative results of thermal stability for various layered structures.

<table>
<thead>
<tr>
<th>Annealing temperature</th>
<th>W/Si</th>
<th>Al/W/Si</th>
<th>Al/WNₓ/W/Si (N₂ plasma 100 W)</th>
<th>Al/WNₓ/W/Si (N₂ plasma 200 W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>500 °C</td>
<td>X*</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>550 °C</td>
<td>X</td>
<td>WAl₁₂</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>575 °C</td>
<td>X</td>
<td>WA₁₂-, WA₁₃</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>600 °C</td>
<td>X</td>
<td>WA₁₂-, WA₁₃, W(AlSi)₂_, WSi₂-W₁₂, W₁₂</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>650 °C</td>
<td>WSi₂</td>
<td>W₁₂, W(AlSi)₂, WSi₂</td>
<td>W₁₂, W(AlSi)₂</td>
<td>W₁₂, W(AlSi)₂</td>
</tr>
</tbody>
</table>

*“X” indicates no observation of compound phase.

![Image](image-url)

**Fig. 9.** Histograms showing the distributions of reverse bias leakage current density measured at -5 V for (a) Al/p⁺-n, (b) Al/W(100 nm)/p⁺-n, and (c) Al/W(450 nm)/p⁺-n junction diodes annealed at various temperatures.

**D. SEM observation**

Scanning electron microscopy (SEM) was used to investigate the surface and cross-sectional morphologies of the Al/barrier/p⁺-n junction diodes. Figure 12 shows SEM micrographs of the Al/W(450 nm)/p⁺-n junction diodes before and after thermal annealing. The as-deposited samples have rough Al surfaces because of the rough surfaces of the CVD-W underlayers [Fig. 12(a)]. Both the surface morphology and the W/Si interface remained unchanged after annealing at 500 °C [Fig. 12(b)]. After annealing at 600 °C, precipitates were found at the W/Si interface and protrusions

![Image](image-url)

**Fig. 10.** Histograms showing the distributions of reverse bias leakage current density measured at -5 V for the W(450 nm)/p⁺-n junction diodes annealed at various temperatures.
were observed on the surface, presumably due to WAl$_{12}$ formation [Fig. 12(c)]. Assuming total conversion of the 500-nm-thick Al film into the WAl$_{12}$, only about 40 nm of W-layer thickness was consumed and the remaining 410 nm should be able to function as a barrier layer. However, non-uniform formation of WAl$_{12}$ might lead to a highly localized thinning of the W layer; thus, either Al atoms or WAl$_{12}$ compound can be very close to the W/Si interface. As a result, close proximity of Al atoms to the Si substrate can easily induce reaction between Al and Si during thermal annealing at 600 °C. Moreover, it is possible that the formation of WSi$_2$ and WAl$_{12}$ introduces microcracks that cannot be observed by SEM but might provide fast diffusion paths for Al atoms to react with the Si substrate.

Figure 13 shows top-view (left) and cross-sectional view (right) SEM micrographs for (a) 600 °C annealed Al/WN$_x$/W(450 nm)/$p^+$-$n$ junction diodes whose W barrier was treated with N$_2$ plasma at 50 W, (b) 600 °C annealed Al/WN$_x$/W(450 nm)/$p^+$-$n$ junction diodes whose W barrier was treated with N$_2$ plasma at 100 W, and (c) 650 °C annealed Al/WN$_x$/W(450 nm)/$p^+$-$n$ diodes whose W barrier was treated with N$_2$ plasma at 100 W.

![Histograms showing the distributions of reverse bias leakage current density measured at −5 V for the Al/WN$_x$/W(450 nm)/$p^+$-$n$ junction diodes annealed at various temperatures; the W layers of the junction diodes were treated with N$_2$ plasma at (a) 50, (b) 100, and (c) 200 W.](image)

![Top-view (left) and cross-sectional view (right) SEM micrographs for the Al/W(450 nm)/$p^+$-$n$ junction diodes (a) as-deposited, (b) annealed at 500 °C, and (c) annealed at 600 °C.](image)
on the W surface due to the N₂ plasma treatment, was able to suppress the WAl₁₂ compound formation and kept the W/Si interface unchanged up to 600 °C. After annealing at 650 °C, all of the diodes failed, presumably due to the low melting point of Al (660 °C). Compound WAl₁₂ and WAl₆, and W(Si, Al)₂ formation along with tungsten silicidation deteriorated the Al/WNₓ/W(450 nm)/p⁺-n diodes completely, as shown in Fig. 13(c). Comparative thermal stability results for the p⁺-n junction diodes with different W-barrier layers determined using various characterization techniques are given in Table II.

### E. SIMS analysis

SIMS depth profiles were used to examine elemental distributions in the multilayer structures. Figure 14 shows the SIMS depth profiles for the Al/W(450 nm)/Si and Al/WNₓ/W(450 nm)/Si samples before and after thermal annealing at 600 °C. Without N₂ plasma treatment, the interdiffusion of Al, W, and Si led to severe deterioration of the Al/W(450 nm)/Si structure after 600 °C annealing, as shown in Fig. 14(b). The Al/WNₓ/W(450 nm)/Si samples whose W barriers were treated with N₂ plasma at 200 W, had W and Si profiles that remained almost unchanged, while Al diffused only slightly into W layers, as shown in Fig. 14(d). This indicates that the thermal stability of the Al/W barrier/p⁺-n diodes can be significantly improved using N₂ plasma treatment on the W surfaces.

### IV. SUMMARY

The effect of in situ N₂ plasma treatment on the thermal stability of selective CVD-W contacted p⁺-n junction diodes with Al metallization was systematically investigated. Without N₂ plasma treatment, the Al/W(100 nm)/p⁺-n and Al/W(450 nm)/p⁺-n junction diodes were able to sustain a 30 min furnace annealing up to 500 and 575 °C, respectively. With in situ N₂ plasma treatment on W surfaces at a power of 100 W, the Al/WNₓ/W(450 nm)/p⁺-n junction diodes were able to sustain thermal annealing up to 625 °C without degradation of their electrical characteristics. Reaction between Al and CVD-W at elevated temperatures resulted in formation of WAl₁₂, WAl₆, and W(Si, Al)₂, leading to acceleration of the reaction between W and Si substrates. The N₂ plasma treatment resulted in formation of a thin WNₓ layer on the W surface, and the Al/WNₓ/W/Si structure effectively suppressed W-Al compound formation, leading to improvement of thermal stability.

### ACKNOWLEDGMENT

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Table II. Comparative thermal stability results for p⁺-n junction diodes with different W-barrier layers as determined using various characterization techniques.

<table>
<thead>
<tr>
<th>Barrier/metallization structures of p⁺-n junction diodes</th>
<th>Leakage current</th>
<th>SEM</th>
<th>SIMS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al/W(100 nm)/p⁺-n</td>
<td>500 °C</td>
<td>500 °C</td>
<td></td>
</tr>
<tr>
<td>Al/W(450 nm)/p⁺-n</td>
<td>575 °C</td>
<td>575 °C</td>
<td>&lt;600 °C</td>
</tr>
<tr>
<td>Al/WNₓ/W(450 nm)/p⁺-n (N₂ plasma at 200 W)</td>
<td>625 °C</td>
<td>625 °C</td>
<td>&gt;600 °C</td>
</tr>
<tr>
<td>Al/WNₓ/W(450 nm)/p⁺-n (N₂ plasma at 200 W)</td>
<td>625 °C</td>
<td>625 °C</td>
<td>&gt;600 °C</td>
</tr>
<tr>
<td>W(450 nm)/p⁺-n</td>
<td>700 °C</td>
<td></td>
<td>WSi₂ formed at 700 °C</td>
</tr>
</tbody>
</table>

Fig. 14. SIMS depth profiles for Al/W(450 nm)/Si (a) as-deposited and (b) 600 °C annealed, and for Al/WNₓ/W(450 nm)/Si (c) as-deposited and (d) 600 °C annealed.

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