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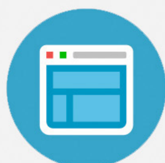

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Optimization of post-N_2 treatment and undoped-Si-glass cap to improve metal wring delamination in deep submicron high-density plasma-fluorinated silica glass intermetal dielectric application

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Integration issues of metal line delamination from fluorinated silica glass (FSG) in deep submicron intermetal dielectric applications were investigated in this study. A metal line peeled off after a nonoptimized *in situ* deposition of undoped-silicon-glass (USG; SiO_2) capping layer followed the post-FSG-chemical mechanical polishing N_2 treatment. It was found that higher bias power and longer process time of N_2 treatment led to more active fluorine species diffusing from the FSG films to the USG surface, which might react with subsequent Ti/TiN/W metal layer and result in metal delamination. Using plasma-enhanced N_2 treatment and *ex situ* USG capping with lower initial deposition temperature by extra cooling step, the stability of the FSG films was improved and resulted in a robust structure without metal peeling. © *2004 American Vacuum Society.*
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I. INTRODUCTION

As feature size shrinks continuously to deep submicron regime, low-*k* dielectric materials are adopted into damascene structures to reduce interconnection delay and increase device speed.[1,2]

Fluorinated silica glass (FSG) films deposited by high-density plasma chemical vapor deposition (HDP-CVD) have been implemented in advanced intermetal dielectric (IMD) application and mass fabrication. The FSG film has the advantages of low permittivity ($k=3.5-3.8$), high gap-filling capability, and especially simplicity of IMD integration.[3,4] However, high fluorine concentration of the FSG films, exceeding 5 at. %, induces water absorption and degrades film properties.[5–7] Moreover, fluorine outgassing results in metal delamination due to the degradation of adhesion ability.[8,9] Several methods have been proposed to prevent the metal delamination.[10–14] N_2 treatment and capping of undoped-silicon-glass (USG; SiO_2) film on FSG layers have been demonstrated to eliminate the adhesion problem between FSG and Ti/TiN/AlCu/TiN metal stack.[4,8,10] However, the effect of process conditions of N_2 treatment and capping layers on the metal delamination is not fully examined in previous studies.

In this work, the optimum conditions of N_2 treatment and SiO_2 capping layers on the metal delamination were characterized and the mechanism of metal delamination was established as well.

II. EXPERIMENT

FSG films were deposited on a *p*-type 8 in. wafer by an Ultima HDP-CVD Applied Materials Centura 5200 system using SiH_4, SiF_4, O_2, and Ar as precursors. The subsequent N_2 treatment and USG capping layer deposition were carried out in the same chamber without vacuum breaking. These two processes were performed on FSG films prior to undergoing the FSG chemical mechanical polishing (CMP) process. The N_2 treatment was biased by radio-frequency (rf) power. The precursors for the deposition of USG layer were SiH_4 and O_2. The fluorine concentration of FSG films was controlled at 4.5 at. % by the Si–F/Si–O ratio based on Fourier transform infrared spectroscopy (FTIR) and by the calibration with Rutherford backscattering. FTIR spectra were used to measure the peak intensity of the Si—F bonding. FTIR analysis was performed at a resolution of 4 cm^{-1} and averaged 16 scans. Absorption spectra were collected in reflectance mode using a FTIR spectroscope Bio-Rad Win-IR PRO. The Si–F peak height was measured before and after annealing test. This annealing process was carried out in a furnace at 425 °C for 2 h in a N_2 ambient.

The fluorine concentration of the USG capping layer surface (<20 Å) was analyzed by dynamic secondary ion mass spectrometer [time of flight-secondary ion mass spectrom-

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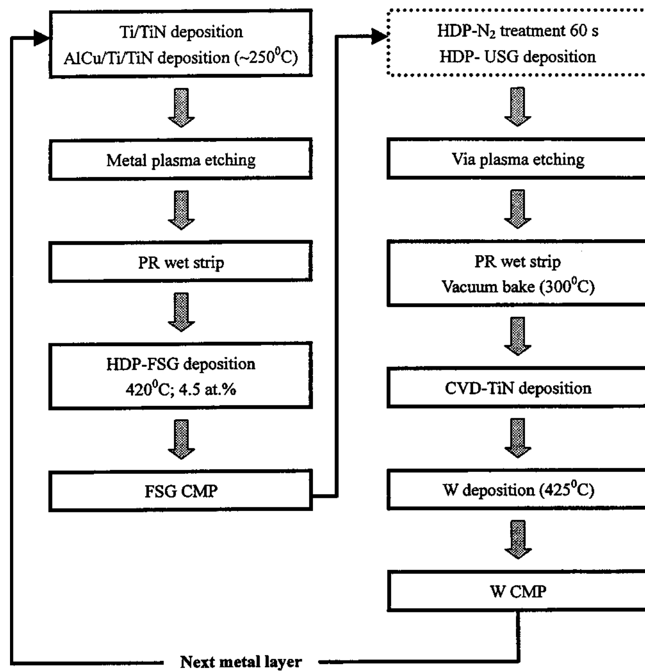

FIG. 1. Repeating process flow for Al/Cu metallization.

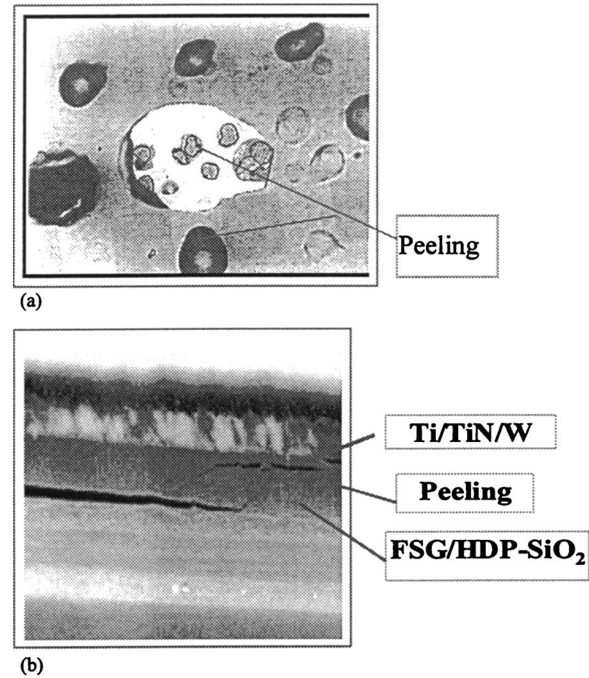

FIG. 2. (a) OM and (b) SEM images of metal delamination.

etry (TOF-SIMS)] with a gallium liquid metal ion gun as the primary ion source. TOF-SIMS was operated in an ion microprobe mode, in which the bunched pulsed primary ion beam was raster scanned across the sample surface. Surface morphologies and cross-sectional images were examined using an optical microscope (OM) and a field emission scanning electron microscope.

III. RESULTS AND DISCUSSION

Figure 1 shows the repeating process flow of Al/Cu metallization in sub-0.18 μm scale device technologies, in which the Ti/TiN metals and HDP-FSG films were used as diffusion barrier and dielectric layers, respectively. After FSG-CMP, the subsequent N_2 treatment and USG capping layer deposition were carried out in the same chamber without vacuum breaking. The bias power, process time, and N_2 flow rate of N_2 treatment were 400 W, 60 s, and 300 sccm, respectively. The initial and process temperatures of the SiO_2 deposition were $\sim$380 °C and $\sim$350 °C, respectively. Adopting the above process condition, metal delamination commonly occurred after W-plug deposition. The optical and scanning electron microscopy (SEM) cross-sectional images in Fig. 2 indicate that peeling appeared at the interface between the TiN barrier and FSG/SiO_2.

A. Effect of N_2 treatment on metal delamination

In order to understand the effect of N_2 treatment on the fluorine concentration at the surface of the USG capping layer, a process comparison was implemented. A $\sim$18 kÅ HDP-FSG layer was directly deposited on a silicon substrate and followed by CMP until the thickness of the FSG films reached $\sim$6 kÅ. Then various N_2 plasma treatment conditions were performed and capped with $\sim$2 kÅ USG film. The fluorine concentration at the USG surface was found to be

TABLE I. Normalized fluorine concentration by TOF-SIMS analysis and metal delamination incidence for various N_2 treatment conditions.

Conditions			TOF-SIMS	Delamination
N_2 treatment	Capping layer	Annealing	(F)	results
400 W/60 s	*In situ* HDP-USG dep.	No	191.0	Peeling
400 W/45 s	*In situ* HDP-USG dep.	No	156.2	Peeling
400 W/30 s	*In situ* HDP-USG dep.	No	129.9	Peeling
400 W/10 s	*In situ* HDP-USG dep.	No	100.5	Slight peeling
350 W/60 s	*In situ* HDP-USG dep.	No	102	Peeling
300 W/60 s	*In situ* HDP-USG dep.	No	64.6	Slight peeling
250 W/65 s	*In situ* HDP-USG dep.	No	38.5	No peeling
400 W/60 s	*In situ* HDP-USG dep.	Yes	138.0	Peeling
PE-N_2 (300 W/60 s)	*In situ* HDP-USG dep.	No	33.6	No peeling

Note: dep.=deposition.

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TABLE II. FTIR results and metal delamination incidence for various USG capping conditions.

Conditions			FTIR Δ Si–F peak height	Initial USG dep. temperature (°C)	Delamination results
N_2 treatment	Capping layer	Annealing			
400 W/60 s	*In situ* HDP-USG dep.	No	0.105	~380	Peeling
400 W/60 s	*In situ* HDP-USG dep.	Yes	0.204	~380	Peeling
400 W/60 s	Pump 20 s+ *In situ* HDP-USG dep.	Yes	0.058	~360	Slight peeling
400 W/60 s	Cooling 10 s+ *In situ* HDP-USG dep.	Yes	−0.082	~340	No peeling
400 W/60 s	Cooling 30 s+ *In situ* HDP-USG dep.	Yes	−0.088	~320	No peeling
400 W/60 s	*Ex situ* HDP-USG dep.	Yes	−0.116	~310	No peeling
300 W/60 s	*In situ* HDP-USG dep.	Yes	−0.095	~320	No peeling
PE-N_2 (300 W/60 s)	*Ex situ* HDP-USG dep.	Yes	−0.121	~310	No peeling

Note: dep. = deposition.

strongly dependent on the conditions of N_2 treatment, as shown in Table I, in which the normalized fluorine concentration means the 100-fold ratio of the fluorine concentration to the ^{28}Si concentration. Higher bias power or longer process time of N_2 treatment caused more fluorine content at the USG surface. It was also found that the bias power of N_2 treatment dominated the treatment time in affecting the surface fluorine concentration of USG. At the same N_2 treatment time of 60 s, the higher bias power of 400 W led the surface fluorine concentration of USG to be 195% higher than that for the lower bias power of 300 W, while the bias power was controlled at 400 W, the longer treatment time of 60 s induced the surface fluorine concentration of USG only 47% higher than that for the shorter treatment time of 30 s.

Table I also summarizes the relationship between the various conditions of N_2 treatment and the incidence of the metal delamination. It was found that higher bias power and longer process time of N_2 treatment generated higher fluorine concentration at the USG surface resulting in a higher probability of metal peeling. The results indicated that the higher bias power and longer process time of N_2 treatment produced more actively fragmented fluorine species from the FSG films,[15] and the unstable fluorine species diffused to the USG surface and then reacted with a metal-stacked layer in a subsequent thermal process. Furthermore, the surface fluorine concentration of USG decreased after annealing, however, the improvement of metal delamination was limited and peeling was still observed. The plausible reason was that the annealing process accelerated the unstable fluorine species to react with the TiN/Ti/Al layers leading to another type of peeling.

Although the reduction of bias power and process time of N_2 treatment effectively reduced the surface fluorine concentration of USG, the metal line delamination randomly occurred in the processes. During the N_2 treatment with rf power, a negative voltage was exerted on the back side of the processing wafer; then the N_2 plasma had a higher driving force to break the Si—F bonds of FSG films. Therefore, a feasible treatment needs to substitute for rf bias power to improve the stability of FSG films. As shown in Table I, the adoption of plasma-enhanced N_2 (PE-N_2) treatment significantly decreased the surface fluorine concentration of USG resulting in the elimination of the metal delamination.

B. Effect of capping conditions on metal delamination

The FTIR Si–F peak intensity of the FSG films was found to be dependent on the initial deposition temperature of USG capping layers as shown in Table II. Figure 3 shows the evolution of initial deposition temperature in different capping conditions, in which the cooling or pumping step inserted between the N_2 treatment and USG deposition was used to decrease the initial capping temperature. The Si–F peak intensity of the FSG surface was measured before and after the annealing processes. The Si–F peak intensity increased after annealing when the initial deposition temperature of USG was estimated about 370–380 °C. A slightly increase of the Si–F peak height after annealing was found when the initial capping temperature was about 360 °C. In contrast, when the initial capping temperature was 310–320 °C, the Si–F peak height decreased after annealing. Interestingly, the occurrence of metal delamination showed a

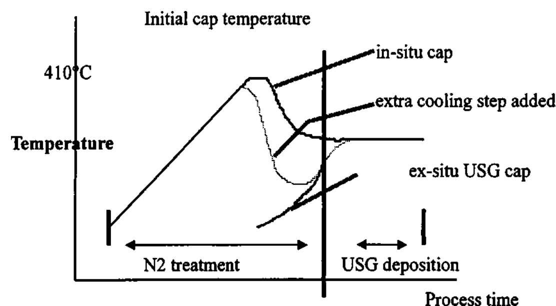

FIG. 3. Thermal evolution of substrates for different USG capping conditions.

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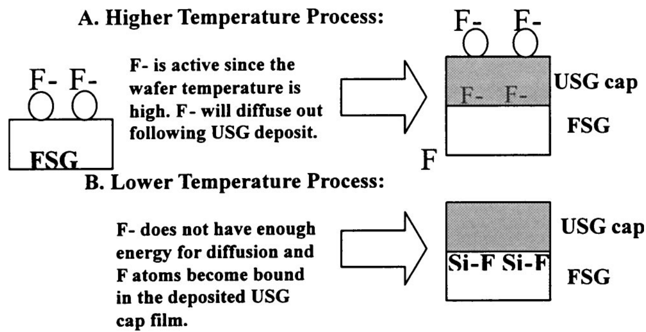

FIG. 4. Proposed mechanism of fluorine diffusion under different initial deposition temperature of USG capping layers: (a) higher-temperature process and (b) lower-temperature process.

strong correlation with the initial deposition temperature of USG. When the temperature was above 360 °C, serious peeling occurred on metal pads.

It was suggested that the active fluorine species in the FSG films resulted from high bias power and long process time of N_2 treatment, or high initial deposition temperature of USG capping layers not only formed the Si–F bonds within the film, but also diffused to the USG surface, as shown in Fig. 4. Therefore, when the initial capping temperature of USG was higher (>380 °C), the trapped fluorine at the USG surface further induced the metal delamination after the subsequent metallization process. A secondary ion mass spectrometry (SIMS) profile of a FSG/USG stacked film with a higher initial USG deposition temperature proves the hypothetical mechanism (Fig. 4) shown in Fig. 5. As can be seen, the SIMS profile shows that a sharp fluorine peak occurred at the FSG/USG interface and a high fluorine concentration was present within the USG capping layer. The result indicates that fluorine would accumulate in the FSG/USG

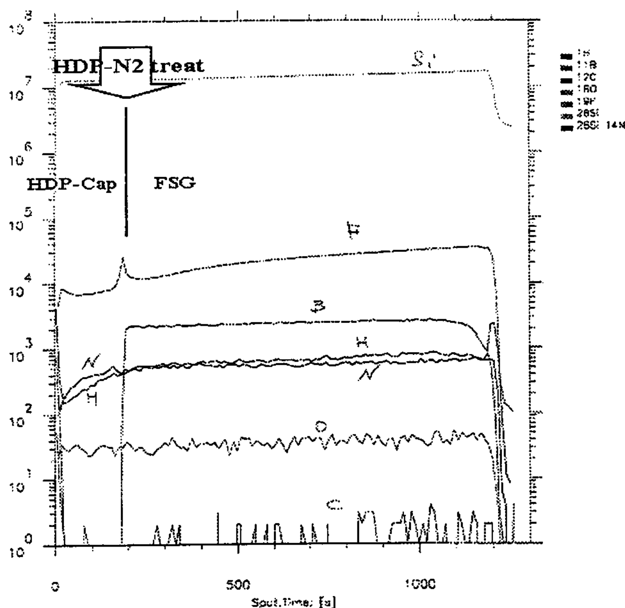

FIG. 5. SIMS depth profile of the FSG film with *in situ* HDP-N_2 treatment and HDP-USG capping layer.

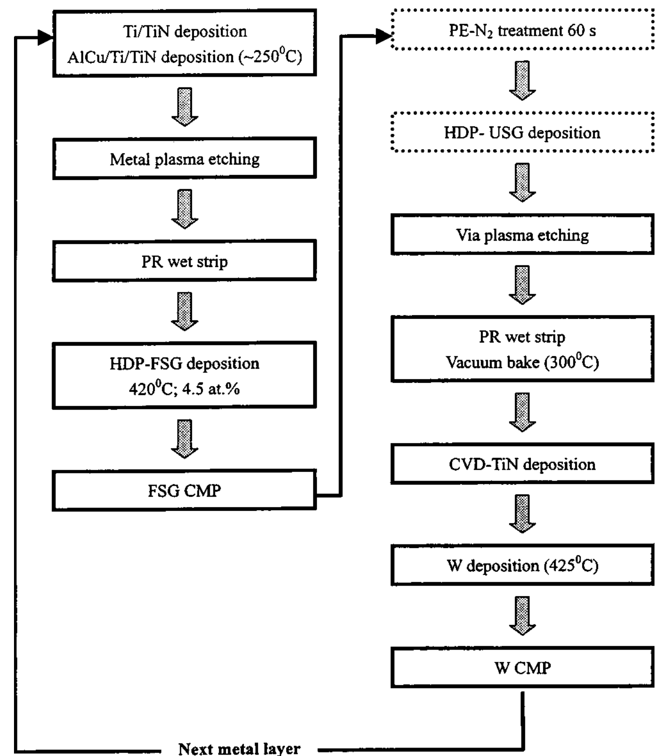

FIG. 6. Optimized process flow for Al/Cu metallization.

interface and diffuse into the USG layer during higher USG deposition temperature. This higher fluorine content within the USG layer has the potential risk of resulting in metal delamination after the subsequent metallization process.

On the other hand, when the initial deposition temperature of USG was as low as 320 °C, the driving force was not enough for unbonded fluorine to diffuse out. However, free fluorine atoms bonded with Si to form the Si—F bond by the thermal energy at the beginning of USG deposition. The fluorine source was exhausted in the USG/FSG surface resulting in no extra FTIR Si–F peak at the USG/FSG interface. As a result, the Si–F peak height decreased due to no additional fluorine to form Si—F bonds in the following annealing process.

Moreover, it was found that using *ex situ* USG deposition significantly decreased the Si–F peak after annealing (see Table II) when the initial deposition temperature of USG was reduced to 300 °C. As expected, this separated USG deposition method resulted in no metal line delamination.

To prevent the metal delamination, a modified process flow was proposed as shown in Fig. 6. PE-N_2 treatment and a separate USG capping process with an initial deposition temperature below 350 °C were used to broaden the process window. Based on this metallization process, a robust structure without metal peeling was obtained. Furthermore, the SIMS profile of Fig. 7 shows that lower fluorine concentration was present in the USG film as compared with the result of Fig. 5.

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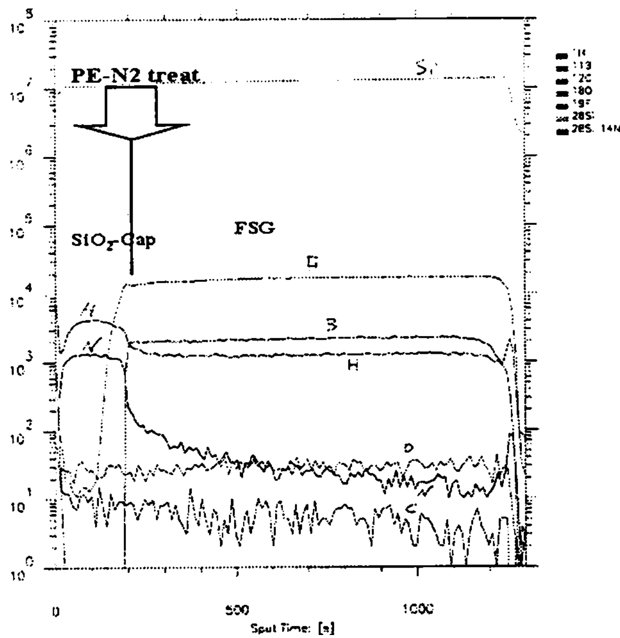

FIG. 7. SIMS depth profile of the FSG film with PE-N_2 treatment and *ex situ* HDP-USG capping layer.

IV. CONCLUSIONS

Fluorine stability is extremely important in HDP-FSG IMD application. In this study, the SIMS and FTIR results showed that high rf-bias power and long process time of N_2 treatment or highly initial deposition temperature of USG capping layers generated active fluorine species from the FSG films. The unbonded fluorine species diffused to the USG surface and reacted with the subsequent Ti/TiN layer resulting in metal delamination. PE-N_2 treatment and a separate USG capping with the initial deposition temperature below 350 °C by an extra cooling step were found to be useful to prevent metal delamination.

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