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Abstract

In this study, hydrogen silsesquioxane (HSQ) thin films prepared under various conditions are employed as the intermetal dielectric and the high frequency characteristics of Al/HSQ system are investigated and compared with those of $Al/SiO₂$ system. The *S*-parameters of the Al interconnect are measured for insertion loss and crosstalk noise. A figure of merit (FOM) is employed to evaluate the characteristics of the Al/HSQ system at high frequencies (100MHz \sim 20GHz). It is found that Al interconnect with HSQ films annealed at 400° C has an insertion loss of 1.64dB/mm, a coupling of -13.32 dB at 20GHz, while those of the PECVD SiO₂ films are 2.01dB/mm (insertion loss) and -13.40 dB (coupling). The Al- 400° C-annealed-HSQ system has better performance than the $AI-SiO₂$ system does from 100MHz to 20GHz. However, specimens with 350°C-annealed HSQ films or plasma-treated HSQ films exhibit larger insertion losses and higher crosstalk noises than those with PECVD $SiO₂$ films do. Both annealing temperature and O_2 plasma treatment of the HSQ films affect the high frequency characteristics of the Al/HSQ system. Besides, implementation of Cu/HSQ is used for reducing insertion loss, RC delay, crosstalk noises, etc. In this work, Cu/hydrogen silsesquioxane (HSQ) systems are studied, too. Ammonia (NH3) plasma is employed for the nitridation of HSQ. The effects of $NH₃$ plasma treatments on the high frequency characteristics (100 MHz to 20 GHz) of the interconnect structure Cu/Ta/HSQ. The smallest insertion loss is 1.97 dB/mm at 20 GHz for the 400 \degree C-annealed Cu/Ta/HSQ (NH₃-plasma-treated for 50 sec). Appropriate NH₃-plasma bombardment helps to form a thin $\sin X_x$ barrier layer which prevents the diffusion of oxygen without increasing the dielectric constant of the Cu/HSQ interconnect system.

Keywords: *S*-parameters, high speed interconnect, hydrogen silsesquioxane, insertion loss, low *k* dielectric, crosstalk

Introduction

As the dimensions of ULSI circuits shrink, there is a need for faster performance and higher circuit density. Multilayer interconnect structures are the trend to produce high-density circuits and enhance device performance. However, at high operational frequencies (>1GHz), the parasitic effects associated with the multilayer interconnect become a limiting factor for chip performance and can not be ignored [1-3].

The parasitic effects of the multilayer interconnect are comprised of resistance with variation due to the skin effect at high frequencies, the self- and mutual-inductance of interconnects, the shunt capacitances from signal lines to ground lines, the parasitic capacitance resulted from the intermetal dielectric, the leakage of the dielectric material and substrate…etc. All result in signal distortion, propagation delay, and crosstalk noise [3-9]. In order to retain signal integrity at high frequencies, the requirements for the intermetal dielectric include: a small dielectric constant, low leakage current, low moisture absorption, adequate mechanical strength, simplicity of process, and ease of integration [10-15].

Hydrogen silsesquioxane (HSQ) is a potential candidate of which the dielectric constant can be further reduced by forming a highly porous three-dimensional network structure [14]. The general formula for HSQ is $(HSiO_{1.5})_{2n}$, n= 2, 3, etc., which is an inorganic material that can be considered as a derivative of $SiO₂$ in which one of the four oxygen atoms bonded to every silicon atom is replaced by hydrogen. However, HSQ has many integration issues, such as: thermal dissociation of Si-H bonds, oxidation, plasma damage, formation of -OH bonds, absorption of water, etc. [11-13]. It was reported that with appropriate plasma treatments the thermal stability of HSQ could be enhanced [12]. Nevertheless, the plasma treatment may damage the surface and/or change the surface chemistry of HSQ and the reliability of the low *k* dielectric HSQ may degrade [11-13].

In this work, HSQ films are prepared and plasma treatments are applied to some specimens. The electrical properties of Al interconnect on HSQ and/or $SiO₂$ aare measured and compared. The high frequency characteristics of the Cu/Ta/HSQ systems are studied, too. Finally, the interconnect systems with and without embedded process are compared.

Experimental Procedures

Four inch diameter p-type (100) Si wafers with nominal resistivity of 5 to 10 Ω-cm were used as substrate. After standard RCA cleaning, a $100 \text{nm } \text{SiO}_2$ film was grown on the Si substrate. Two dielectrics, hydrogen silsesquioxane (HSQ) and $SiO₂$, were deposited on top of the $SiO₂/Si$ substrate. Hydrogen silsesquioxane (HSQ) was prepared by spin-coating Dow-Corning Flowable Oxide (FOX) on the wafer and then baked at 150°C, 250°C, and 350°C for 1 minute. Annealing was performed in N_2 furnace from 350°C to 400°C for 60 minutes as described in [13]. The thickness of HSQ is about

500nm after annealing. Some samples were subjected to plasma treatment. The $O₂$ plasma was operated at a pressure of 40Pa for 5 minutes, a plasma power of 100W and a chamber temperature of 250 $^{\circ}$ C. The amorphous $SiO₂$ films, deposited by the decomposition of tetraethyl orthosilicate, with 500nm in thickness were deposited with PECVD (Multichamber PECVD, STS-MULTIPLEX CLUSTER SYSTEM, England) at 300°C (substrate temperature) and 200W. Then, 500nm Al films were deposited by thermal evaporation onto the dielectric material to serve as interconnect. On the other hand, for Cu interconnect system, the curing condition for HSQ films is 400° C for 1 hour in N_2 . Some samples were subjected to NH_3 plasma treatment for different durations. The NH3 plasma was operated at a pressure of 300mtorr and with a flow rate of 500sccm in a PECVD chamber. An rf power of 200W, which established the NH3 plasma, was applied to the HSQ films at a substrate temperature of 300° C and a chamber temperature of 250°C. For *S*-parameter measurements, a Cu/Ta/HSQ/SiO₂/Si multilayer structure was used and conventional lithographic processes were employed to obtain the interconnect patterns. Some samples were annealed at 400° C for 1 hour in N2 at a pressure of 10torr before *S*-parameter measurements.

The imizidation and chemical bond structure of HSQ films were investigated by Fourier transform infrared (FTIR) spectrometer (DA8.3, Bomen Inc., Canada). A C-V analyzer (model 590, Keithley Instruments Inc., U.S.A.) and a semiconductor parameter analyzer (HP4155B, Hewlett Packard Co., U.S.A.) were employed to measure the capacitance and the leakage current, respectively. A Network Analyzer (HP-85122A, Hewlett Packard Co., U.S.A.) which operates from 100MHz to 20GHz was used to measure the *S*-parameters of the interconnect on-wafer with the microwave probes. Before each measurement, the calibrations were carried out with SOLT pads (short, open, load, and through) purchased from Cascade Microtech (Cascade Microtech, U.S.A.). A de-embedding method proposed in [16] was employed to deduct the shunt parasitics of the probing pads. Then, the signal transient characteristics with signal delays and crosstalk noises between adjacent interconnect lines are evaluated by the inverse Fourier transform from *S*-parameters of the interconnect.

Results and Discussion

A typical structure of HSQ is an Si-O cage containing Si-H bonds. The three-dimensional network structure of HSQ is obtained through annealing process. After annealing, some of the Si-H bonds dissociate and the cage is rearranged into network structure as shown schematically in Fig. 1(a) [13, 14]. Fig. 1(b) shows the Fourier transform infrared (FTIR) spectra before and after annealing with or without $O₂$ plasma treatment. The spectra exhibit a Si-H peak at 2250cm^{-1} , Si-O stretching cage-like peak at 1130cm^{-1} , Si-O stretching network peak at 1070cm^{-1} , Si-O bending cage-like peak at 863cm^{-1} and Si-O bending network peak at 830cm^{-1} as listed in Fig. 1(a) [11]. Datum in the brackets in Fig. 1(b) is the relative Si-H peak area with respect to the Si-H peak area of the as-deposited HSQ films. The Si-H bond (2250cm^{-1})

absorbance decreases as annealing temperature increases especially after $O₂$ plasma treatment. The Si-O stretching cage-like bond (1130cm^{-1}) and Si-O bending cage-like bond $(863cm⁻¹)$ break to form Si-O stretching network bond $(1070cm⁻¹)$ and Si-O bending network bond (830cm^{-1}) , respectively.

The test structure for insertion loss measurement has one signal line as exhibited in Fig. 2(a). With the ground-signal-ground probes, insertion loss (dB/mm) is obtained from the *S*-parameters $S21_s$ and $S11_s$ where the subscript s denotes one signal line. Insertion $loss = 10 \times log[$ $||S21_s|^2/(1 - |S11_s|^2)]$ / length (dB/mm)

Specimens with 400°C-annealed HSQ dielectric have the smallest insertion loss, i.e., the largest $S21_s$, among all the specimens studied. However, O_2 plasma treatment increases the insertion loss of the interconnect, as shown in Fig. 2(b). Specimens with 400°C-annealed HSQ have the smallest insertion loss, however, plasma treatment raises the conductance of the 400° C-annealed HSQ. Our work [13] indicates that exposure to the O_2 plasma causes Si-H bonds to break. The breaking of Si-H bonds leads to the formation of dangling bonds which absorb water rapidly to form Si-OH bonds when the HSQ is exposed to air after the O_2 plasma treatment [13].

Crosstalk noises between two interconnect lines are measured with the test structure shown in Fig. 3(a) where signal is applied to the aggressor line (S1) and coupling voltage across the victim line $(S2)$ is measured. The *S*-parameter $(S21_d)$ is employed to evaluate the coupling between the two lines. As exhibited in Fig. $3(b)$, $S21_d$ increases with the increase of frequency, and specimens with the 400° C-annealed HSQ films have the lowest $S21_d$ at frequencies below 15GHz. While those with 350° C-annealed HSQ films and 400° C-annealed HSQ films followed by O_2 plasma treatment show less coupling at higher frequencies (>15GHz). Increasing the spacing/width ratio of the interconnect decreases the coupling as seen in the inset of Fig. 3(b). The transient voltages of the victim line are evaluated by the inverse Fourier transform when a step voltage is applied to the aggressor interconnect. As exhibited in Fig. 4(a), specimens with 400°C-annealed HSQ films have a smallest transient peak (i.e., crosstalk noise) of 0.121V. The transient peaks are 0.136V, 0.136V, and 0.130V, for specimens with 350°C-annealed HSQ films, 400°C-annealed HSQ films followed by O_2 plasma treatment, and PECVD $SiO₂$, respectively. The first transient peak is mainly caused by capacitive coupling, and the following oscillations are attributed to both capacitive and inductive coupling.

As mentioned previously, among the specimens studied, samples with 400°C-annealed HSQ have the smallest signal attenuation and crosstalk noise from 100MHz to 15GHz. However, at frequencies above 15GHz, less coupling is observed for specimens with 350° C-annealed HSQ or O_2 plasma-treated HSQ. A figure of merit (FOM) is thus defined to evaluate the high frequency performance of the interconnect on the basis of both signal attenuation and crosstalk noise.

 $FOM = |S21_s| \times (1 - |S21_d|)$

The larger $S21_s$ (the smaller signal attenuation) and the smaller $S21_d$ (the smaller crosstalk), the larger the FOM. As exhibited in Fig. 4(b), specimens with 400°C-annealed HSQ films show the largest FOM over the range of 100MHz to 20GHz among all the specimens studied.

Fig. 5 shows the FTIR-RAS spectra of specimens as a function of $NH₃$ plasma treatment time. Plasma treatment breaks the Si-H, Si-O bending cage-like, and Si-O bending network bonds. The Si-O bending cage-like peak ratio (895cm^{-1}) , the Si-O bending network peak ratio (822cm^{-1}), and the Si-H peak ratio (2250cm^{-1}) decrease with the increase of NH₃ plasma bombardment time, while the Si-N peak ratio (985cm⁻¹) increases with plasma bombardment time. Based on the leakage current mechanisms [17], the slope of the Ln(J) versus $E^{1/2}$ curve, as summarized in Table 1, is β/kT where β is β_{SE} (for Schottky emission) or β_{PF} (for Poole-Frenkel emission). Both β_{SE} and β_{PF} are calculated and listed in Table 1 for comparison. The β values obtained in this study are closer to β_{SE} and this suggests that the dominant mechanism for charge carriers to transport in HSQ is Schottky emission. The barrier height increases from 1.27eV of the as-cured HSQ to a maximum of 1.39eV of the 120sec plasma treated HSQ and then decreases to 1.18eV of the 1800sec plasma treated HSQ.

Fig. $6(a)$ exhibits the insertion loss and the $S21_s$ parameter of various samples, while the test structure is shown in Fig. 2(a). Annealing raises the insertion loss (i.e., decreases *S*21s) of the interconnect. The as-deposited Cu/Ta/HSQ interconnect has the smaller insertion loss, i.e., the larger $S21_s$, than the 400°C-annealed Cu/Ta/HSQ. Plasma treatment decreases the insertion loss of the interconnect. Specimens subjected to 50sec NH₃ plasma bombardment show the smallest insertion loss (the largest $S21_s$) among all specimens in this study. The insertion losses are 2.45dB/mm, 2.44dB/mm, and 1.66dB/mm at 10GHz, and 2.68dB/mm, 2.63dB/mm, and 1.97dB/mm at 20GHz for the as-deposited Cu/Ta/HSQ, the 400°C-annealed Cu/Ta/HSQ, and the 400°C-annealed Cu/Ta/HSQ(NH3-plasma- treated for 50sec), respectively. The specimens whose test structures are exhibited in Fig. 3(a) with the 400°C-annealed Cu/Ta/HSQ $(NH_3$ -plasma-treated for 50sec) show larger $S21_d$ at lower frequency and all specimens show the similar $S21_d$ at higher frequency than 8GHz, as exhibited in Fig. 6(b).

As shown in Fig. 7(a), we compare the series inductance and resistance of the interconnect before the after the passivation layer for Al/HSQ interconnect structure, whose interconnect lengths are 400 μ m and 1000 μ m, respectively. The inductances of the interconnect are stable with and without the embedded, but the resistances of the interconnect increase after the embedded process. It possibly results from the imperfect etching of the via hole. Therefore, the resistances of the interconnect increase after the embedded process. In Fig. 7(b), the capacitors with and without the embedded process are compared. The capacitances increase slightly after the embedded process on HSQ/Si substrate but they are stable as the function of the voltage.

Conclusions

Hydrogen silsesquioxane (HSQ) films prepared under various conditions were integrated with Al interconnects. The dielectric constant of HSQ films annealed at 400°C is smaller than those of HSQ films annealed at 350° C or subjected to O₂ plasma bombardment. The FTIR spectra suggest that absorption of water via open pores or by dangling bonds cause the increase of the dielectric constant. The high frequency characteristics of the Al/HSQ interconnect system are studied and compared to those of the $AI/SiO₂$ system on the basis of the measured *S*-parameters. The AI/HSO system with 400°C-annealed HSQ exhibits smaller insertion loss, lower crosstalk noise, and smaller signal propagation delay than the $AI-SiO₂$ system does.

For the Cu-interconnect system, NH₃ plasma treatment is employed to form silicon nitride on HSQ films to prevent the diffusion of oxygen and oxidation of copper. However, the effect of NH_3 plasma treatment is multiple. Besides forming SiN_x , plasma bombardment breaks the Si-H bonds and result in hygroscopic Si dangling bonds. The barrier height for Schottky emission increases from 1.27eV of the as-cured HSQ to a maximum of 1.39eV of the 120sec plasma treated HSQ and then decreases to 1.18eV of the 1800sec plasma treated HSQ. Among various specimens studied, the smallest insertion loss is 1.97dB/mm at 20GHz for the 400°C-annealed Cu/Ta/HSQ (NH3-plasma-treated for 50sec). Appropriate NH3-plasma bombardment helps to form a thin SiN_x barrier layer which prevents the diffusion of oxygen without increasing the dielectric constant of the Cu-HSQ interconnect system.

Fig.1 (a) Schematic diagram of the structure of hydrogen silsesquioxane (HSQ) and Infrared absorption assignments applicable to HSQ films. (b) FTIR spectra of HSQ films before and after annealing. Datum in the brackets is the relative Si-H peak area with respect to the Si-H peak area of the as-deposited HSQ films.

Fig. 2 (a) Schematic diagram of the interconnect structure for insertion loss measurement (interconnect length is 400 µm, spacing is 100 µm, and width is 50 µm in this case). (b) Insertion loss and *S*-parameter *S*21s as functions of frequency of frequency of specimens with HSQ films annealed at 350 °C (\Box), 400 °C (Δ), and 400°C followed by O_2 plasma treatment (∇), and PECVD Si O_2 films (\odot).

Fig. 3 (a) Schematic diagram of the interconnect structure for crosstalk measurements with two signal (S) lines and two ground (G) lines. The ends of the signal lines are open. Length $l = 400 \mu m$, spacing s1= 90 μ m, 70 μ m, or 50 μ m, s2= 30 μ m, and width w= 50 μ m. (b) $S21_d$ as a function of frequency, and the inset is the frequency dependence of S21d as a function of s1/w ratio of specimens with HSQ films annealed at 350°C (\Box) , 400°C (Δ), and 400°C followed by O₂ plasma treatment (∇), and PECVD SiO₂ films (\odot).

Fig. 4 Transient voltage across the victim line (S2) when a step signal is applied to the aggressor line (S1). (b) Figure of merit (FOM) as a function of frequency of specimens with HSQ films annealed at 350°C (\square), 400°C (\triangle), and 400°C followed by O₂ plasma treatment (∇), and PECVD SiO₂ films (\bigcirc).

Fig. 5 FTIR spectra of HSQ versus NH_3 plasma treatment time, and the inset is the absorbance peak ratio of bond with respect to that of the as-cured HSQ films as a function of NH3 plasma treatment time. The absorbance wavenumbers are 985 cm⁻¹ for Si-N peak (∇), 2250 cm⁻¹ for Si-H peak (Δ), 895 cm⁻¹ for Si-O bending cage-like peak (\square) , and 822 cm⁻¹ for Si-O bending network peak (\blacksquare) .

Fig. 6 (a) Insertion loss and *S*-parameter $S21_s$ as a function of frequency, (b) $S21_d$ as function of frequency of the as-deposited Cu/Ta/HSO (\Box), the 400°C-annealed Cu/Ta/HSO (\Diamond), and the 400°Cannealed Cu/Ta/HSQ(NH₃-plasma-treated for 50 sec) (\bullet).

Fig. 7 (a) The inductance and resistance of the interconnect as function of frequency before and after the passivation. (b) The capacitance and dissipation factor of the capacitor as function of voltage with and without the embedded process.

NH ₃ Plasma treatment time (sec)	Slope $\frac{(\beta/k_BT)}{(m/V)^{1/2}}$	$\frac{\beta(10^{-24}}{Jm^{1/2}/V^{1/2})}$	$\frac{\beta_{SE} (10^{-24})}{J m^{1/2} / V^{1/2}}$	$\frac{\beta_{\rm PF} (10^{-24} \, m^{1/2}/V^{1/2})}$	ϕ_{SE} (eV)
$\bf{0}$	5.31	3.1	4.4	8.7	1.27
10	4.58	2.7	4.3	8.7	1.30
50	5.77	3.4	4.5	9.0	1.33
120	6.71	3.9	4.3	8.7	1.39
600	4.61	2.7	4.3	8.7	1.29
1800	3.25	1.9	4.3	8.5	1.18

Table 1 The slope (β*/k*B*T)* of the Ln(J) versus *E*1/2 plots and the parameters extracted from curve fitting of the Schottky emission model and Poole-Frenkel emission model.

SE emission model: $Ln(J_{SE}/A*/T^2) = (\beta_{SE}E_{1/2} - \phi_{SE})/(k_BT)$ PF emission model: $Ln(J_{PF}/J_0) = (\beta_{PF}E_{1/2}-\phi_{PF})/(k_BT)$

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