



Via-Filling Capability of Copper Film by CVD

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The via-filling properties of chemically-vapor-deposition (CVD) Cu film filled in deep submicrometer vias (diam of 0.11, 0.15 and 0.18 μm) were investigated with respect to various deposition parameters. A superior void-free via-filling by Cu film can be achieved at low temperature, low pressure, and high concentration of precursor species in the gas phase. However, these favorable conditions for void-free via-filling of Cu film also lead to slightly degraded film properties, including higher resistivity, higher content of impurities, degraded adhesion to substrate, and lower deposition rate, as compared with those obtained under the optimal deposition condition. Thus, a trade-off is needed between void-free via-filling and a superior film property. The key to achieve a void-free via-filling is to have a high probability of a re-emission event for the Cu-containing species inside the vias. Using helium (He) as the carrier gas is preferable to using hydrogen (H_2), because the hydrogen reduction reaction would enhance the deposition of Cu, resulting in a decreased probability of a re-emission event and thus degrading the capability of void-free via-filling. In this study, we have achieved void-free filling by CVD Cu film in deep submicrometer 0.11 μm diam vias with an aspect ratio of 9.1 at and below deposition temperatures of 160°C, using a precursor flow rate of 0.4 cm^3/min and at a deposition pressure of 60 mTorr.

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The application of copper to multilevel interconnection offers a number of advantages over the conventional Al-based metallization, including lower bulk resistivity (1.67 $\mu\Omega\text{ cm}$), superior electromigration resistance, and higher resistance to stress-induced voids.^{1,2} To implement copper into metal lines and vias interconnection, the damascene process is developed to cope with the difficult Cu dry etching problem. There are various techniques of copper film deposition, such as chemical vapor deposition (CVD),^{3,4} conventional as well as ionized metal plasma (IMP) physical vapor deposition (PVD),⁵ and electrochemical deposition (ECD) including electroplating and electroless plating;^{6,7} among these, the CVD method has the advantages of superior step coverage and excellent gap filling capability for high aspect ratio vias and trenches,⁴ making it the most promising technique of Cu film deposition for future integrated circuits application. Although the conformal vias/trenches filling by Cu CVD easily results in a seam within the vias/trenches, it was reported recently that vias/trenches could be completely filled with Cu in a bottom-up way resulting in a seam-free filling.⁸ This newly developed technique used iodine as catalytic surfactant to carry out the bottom-up superfilling. Although the Cu ECD combined with the IMP PVD of a thin Cu seed layer and barrier layer provides a viable solution for integrated circuit technologies above 0.25 μm , the technique of CVD is necessary for future device generations because of the requirement of a more stringent conformal and continuous thin barrier as well as conformal and void-free Cu film filling into deep sub-quarter-micrometer vias.⁹ A number of studies on gap-filling using Cu CVD method have been reported regarding feature sizes larger than 0.20 μm .¹⁰⁻¹⁴ In this work, we investigate via filling of CVD Cu films for a feature size of 0.11 μm with an aspect ratio (AR) of 9.1 with respect to various processing conditions, including the effect of carrier gas.

Experimental

Figure 1 shows a schematic diagram of the low pressure CVD system used in this study. The apparatus consists of a reaction chamber (for Cu film deposition), a load-locked chamber (for sample loading/unloading), and a direct liquid injection (DLI) system for precursor delivery. In the reaction chamber, there is a shower-head injector through which the Cu precursor is introduced into the chamber in a stream of carrier gas. Under the injector, there is a substrate susceptor, which can be heated by a resistive heating element up to

a maximum temperature of 400°C. The susceptor is also rotatable for better uniformity of film deposition. The shower-head injector is movable in vertical direction, so that the distance between the injector and the sample can be adjusted. In this study, the susceptor is set to rotate at a speed of 10 rpm, and the injector is set at a position so that it is 2 cm above the susceptor. The side wall of the reaction chamber as well as the precursor injector is kept at a temperature of 45°C by circulation of warm water to prevent Cu deposition of precursor condensation.

The Cu precursor used in this study is Cu(1,1,1,5,5,5-hexafluoroacetylacetonate)trimethylvinylsilane [Cu(hfac)TMVS] with 2.4 wt % TMVS additive, which enhances the stability of the precursor.^{15,16} The liquid Cu precursor is delivered by the DLI system, which is composed of a liquid flow controller (LFC) and a controlled evaporation mixer (CEM). Initially, the liquid precursor is propelled by nitrogen (N_2) gas through the LFC. It is then vaporized in the CEM and mixed with the carrier gas. Helium (He) or hydrogen (H_2) is used as the carrier gas in this study. The precursor-saturated carrier gas is introduced into the reaction chamber through the gas injector for the CVD of Cu films.

In this study, tantalum (Ta) layers were used as the diffusion barrier for the CVD of Cu films. To investigate the film properties of CVD Cu, Cu films were deposited on a blanket Ta layer of 50 nm thickness, which was deposited on thermal oxide (500 nm thickness)-covered Si wafers. A DC magnetron sputtering system with a base pressure of 1.5×10^{-6} Torr was used to reactively sputter deposit the Ta substrate layer at a pressure of 7.6 mTorr using a Ta target (99.99% purity) in a pure Ar gas. To evaluate the via-filling capability of Cu CVD, three sets of vias of different feature sizes were patterned on fluorine-doped silicon glass (FSG) (1 μm thickness)-covered Si wafer. An IMP sputtering system was used to deposit conformal Ta layers of 25 nm thickness into the high AR vias. The diam of the vias are 0.11, 0.15, and 0.18 μm with the AR of 9.1, 6.7, and 5.6, respectively, as shown in Fig. 2.

For CVD Cu films deposition, the blanket Ta coated substrate or the via patterned substrate (with 25 nm thick Ta lining) wafers were loaded into the CVD system. When the pressure of the load-locked chamber reached 10^{-6} Torr, the substrate wafers (together with the substrate holder) were transferred to the reaction chamber for Cu film deposition. Prior to starting the Cu film deposition, the substrate samples were heated to the desired deposition temperature with the He carrier gas flowing at 25 standard cubic centimeters per minute (sccm) and the chamber pressure maintained at a constant pressure of 150 mTorr. Usually, about 1 h was required for the substrate sample to reach the preset temperature. In this study, Cu CVD was

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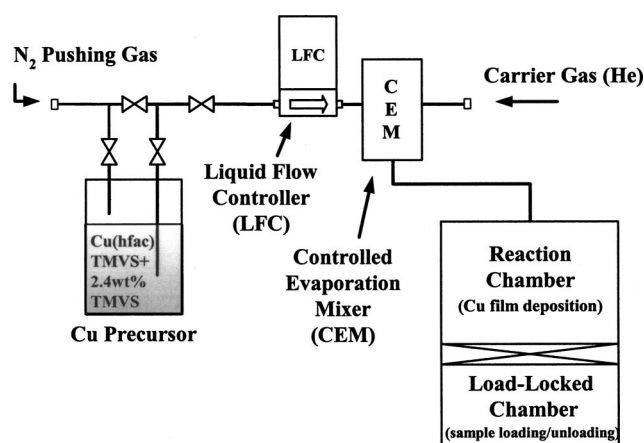


Figure 1. Schematic diagram of low pressure Cu CVD apparatus used in this study.

performed over a temperature range of 120 to 240°C with a precursor flow rate of 0.4 cm³/min at two different pressures and two different carrier gas (He) flow rates, as shown in Table I, unless otherwise specified. At the end of Cu films deposition, the samples were cooled in ambient He at a pressure of 150 mTorr.

The thickness of Cu films was measured using a DekTek profiler on patterned Cu films and was verified by cross-sectional scanning electron microscopy (SEM). SEM was also used to observe the cross-sectional view of Cu-filled vias. A four-point probe was employed to measure the sheet resistance. Auger electron spectroscopy (AES) was used to analyze the impurity content in the Cu films. The Scotch tape pulling test was used to qualify the adhesion between the CVD Cu films and the Ta substrate layers.

Results and Discussion

Properties of CVD Cu films on Ta substrate.—Copper films were chemically vapor deposited on blanket Ta substrate, which has a resistivity of 0.19 mΩ cm. The chemical reaction of Cu-CVD using

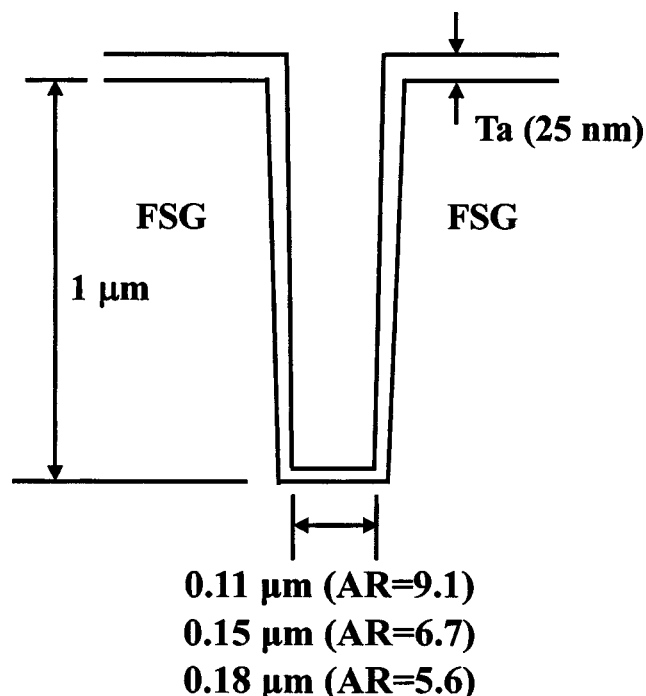
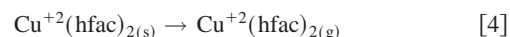
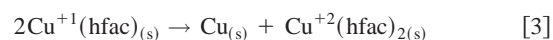
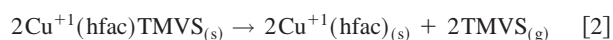


Figure 2. Schematic diagram of deep submicrometer via used in this study.

Table I. Processing conditions for Cu CVD.

Parameters	Case A	Case B
Substrate temperature (°C)	120-240	120-240
Operating pressure (mTorr)	150	60
Carrier gas (He) flow rate (sccm)	25	12
Cu precursor flow rate (cm ³ /min)	0.4	0.4

Cu(hfac)TMVS as a precursor with H₂ as a carrier gas proceeds on the substrate surface by a facile disproportionation as follows^{17,18}



where (g) denotes gas phase and (s) denotes adsorbed on substrate surface. The reaction step 3 (Eq. 3) is the key step of Cu nucleation on the substrate surface, which involves a process of electron exchange between the adsorbed Cu⁺(hfac) and the substrate surface. Thus, it is easier to deposit Cu films on the conducting substrate than the insulating substrate. Since the chemical reaction of Cu CVD involves a thermal dissociation of the Cu precursor (Eq. 2), a higher temperature would result in a higher rate of deposition. For the Cu CVD with H₂ as the carrier gas, a hydrogen-reduction reaction (HRR, Eq. 5) will proceed in parallel with the disproportionation reaction (Eq. 3);¹⁸ this is absent when He is used as the carrier gas. Figure 3 shows the deposition rate of Cu film on Ta substrate as a function of deposition temperature (Arrhenius plot) at two different pressures (conditions of case A and case B, as shown in Table I). The deposition rate of Cu film was calculated using the measured thickness of a Cu film deposited for 10 min. The transition temperature between the surface-reaction-controlled region (at low deposition temperatures) and the mass-flow-controlled region (at high deposition temperatures) is slightly higher for the CVD at low deposition pressure. The activation energy E_a in the surface-reaction-controlled region was determined to be about 10.5 Kcal/mol for both of the different pressure depositions, by the Arrhenius equation

$$R = R_0 \exp(-E_a/kT). \quad [6]$$

where R is the deposition rate, R_0 is known as the Arrhenius pre-exponential constant or frequency factor, k is the Boltzmann constant, and T is the absolute temperature. This value of E_a is very

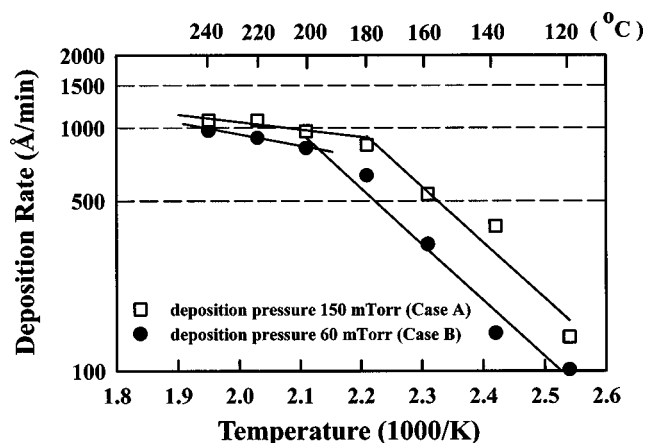


Figure 3. Deposition rate of Cu film vs. deposition temperature (Arrhenius plot) on Ta substrate at two different pressures.

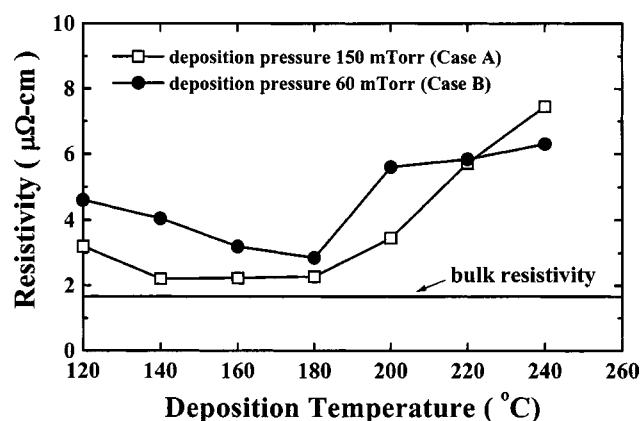


Figure 4. Resistivity vs. deposition temperature for Cu films deposited on Ta substrate at two different pressures.

close to the value of 10.2 Kcal/mol reported in literature.¹⁹ The resistivity of Cu films was calculated using the measured sheet resistance and film thickness. Figure 4 illustrates the resistivity of Cu films as a function of deposition temperature at two different deposition pressures. The resistivity of CVD Cu films is closely related to the impurity content and the films microstructure.^{20,21} The slightly higher resistivity at low deposition temperatures is presumably due to higher contamination of residual impurities from the reaction by-products, while the high resistivity at high deposition temperatures results from the higher contamination of impurities in the film as well as the porous film structure. The generally higher resistivity for the films of Case B is presumably due to the fact that the case B films were deposited using a lower flow rate of carrier gas as compared to the case A films (12 vs. 25 sccm); the carrier gas with a lower flow rate would contain a higher concentration of precursor, and thus resulted in higher contamination of residual impurities from the reaction by-products. Figure 5 shows the AES depth profiles of CVD Cu films; the most notable impurities are carbon (C) and oxygen (O), which arise from incomplete desorption of hfac ligand during the CVD process.²¹ The Cu film deposited with Case B condition reveals a higher content of carbon impurity, which may contribute to the higher resistivity of the film.

A simple technique of Scotch tape pulling test was used to qualify the adhesion between the CVD Cu film and the Ta underlayer substrate, and the results of the adhesion test, with the Cu/Ta system with and without a postdeposition thermal annealing, are summarized in Table II. For the as-deposited Cu films, the Cu films deposited with the case A condition reveal slightly better adhesion than those deposited with the case B condition. It was reported that the impurities (C, F, and O) adsorbed on the substrate surface degraded the Cu film adhesion.^{22,23} Since the carbon impurity content in the Cu film and at the Cu/Ta interface for the films of case B is higher than that of case A (Fig. 5), inferior adhesion of case B Cu

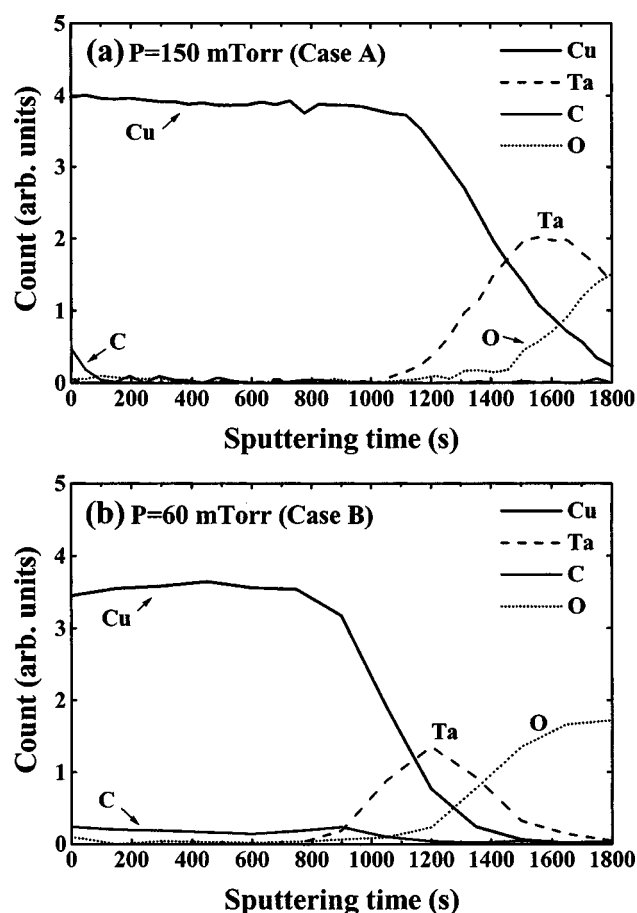


Figure 5. AES depth profiles of Cu films deposited at 160°C on Ta substrate at a pressure of (a) 150 mTorr (case A), and (b) 60 mTorr (case B).

films can be expected. However, low deposition pressure and high precursor content of the case B condition are beneficial to via-filling capability. With a postdeposition thermal annealing at 400°C for 30 min in an N₂ ambient, slight improvement in adhesion was obtained for both case A and case B Cu films.

Via-filling properties of CVD Cu films.—Via-filling characteristics of CVD Cu films was investigated at temperatures ranging from 120 to 200°C under two different sets of deposition conditions, as shown in Table I. At temperature above 200°C, the Cu film deposition belongs to the mass-flow-controlled mechanism, which is disadvantageous to via-filling.¹¹ Post via-filling thermal annealing was performed at 400°C for 30 min in an N₂ ambient to improve the adhesion of Cu films and eliminate the the microvoids between Cu

Table II. Results of Scotch tape pulling test on the adhesion of CVD Cu films to Ta substrate with/without postdeposition thermal annealing (400°C/30 min) for Cu films deposited with case A and case B conditions.

Postdeposition annealing	Processing conditions	Deposition temperatures (°C)							
		120	140	160	180	200	220	240	
No	Case A (P = 150 mTorr)	F ^b	F ^{*c}	P	P ^a	P	P	P	
	Case B (P = 60 mTorr)	F	F [*]	F [*]	P	P	P	P	
Yes	Case A (P = 150 mTorr)	F	P	P	P	P	P	P	
	Case B (P = 60 mTorr)	F	F [*]	F ^{+d}	P	P	P	P	

^a P: Cu film passed the Scotch tape pulling test.

^b F: Cu film peeled off after the Scotch tape pulling test.

^c F^{*}: Cu film partially peeled off after the Scotch tape pulling test.

^d F⁺: Cu film partially peeled off slightly after the Scotch tape pulling test.

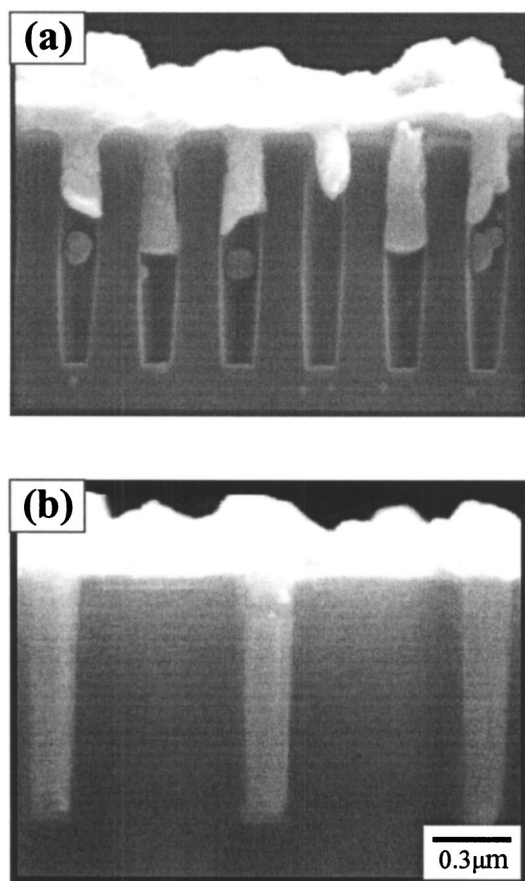


Figure 6. SEM micrographs showing cross-sectional views of via filling for (a) 0.11 μm diameter vias ($\text{AR} = 9.1$) and (b) 0.18 μm diam vias ($\text{AR} = 5.6$) at a deposition temperature of 140°C with case A condition.

grains in the vias. Figure 6 shows the cross-sectional view SEM micrographs of Cu via filling at 140°C under the case A condition. Cu was completely filled in 0.18 μm diam vias, but failed to fill in the bottom of 0.11 μm diam vias. It also failed to achieve a void-free via filling in 0.15 μm diam vias. Figure 7 illustrates the cross-sectional view SEM micrographs of Cu filling in 0.11- μm -diameter vias ($\text{AR} = 9.1$) at various deposition temperatures under case B condition. Void-free Cu filling was achieved at and below the temperature of 160°C (Fig. 7a and b); however, Cu was not able to completely fill in the 0.11 μm vias at higher temperatures of 180 and 200°C (Fig. 7c and d). Cu in the top part of vias (Fig. 7a) was scraped off during the adhesion test of Cu in the vias, indicating inferior adhesion for the Cu films deposited at 140°C. In fact, complete and void-free filling by CVD Cu film in vias of all feature sizes (diam of 0.11, 0.15, and 0.18 μm) was achieved at and below 160°C under case B condition (low pressure and high precursor content in the carrier gas). Comparative results of via filling by CVD Cu film in vias of various sizes deposited at different temperatures for case A and case B conditions are summarized in Table III.

The mechanism as well as the capability of via filling by CVD Cu film is closely related to the deposition condition of Cu films. A precursor species that impinges on a surface can either become adsorbed or be re-emitted from the surface into the gas phase. If a precursor species is adsorbed, it can either nucleate at the point of adsorption or diffuse along the surface. After diffusion, the precursor species can again be either re-emitted from the surface or nucleate at the surface.²⁴ The total reactive sticking coefficient (S_c), which is defined as the total probability that a precursor species will nucleate after impingement on the surface, takes each of these possibilities into account.^{10,25} It was reported that a low S_c (or high probability of

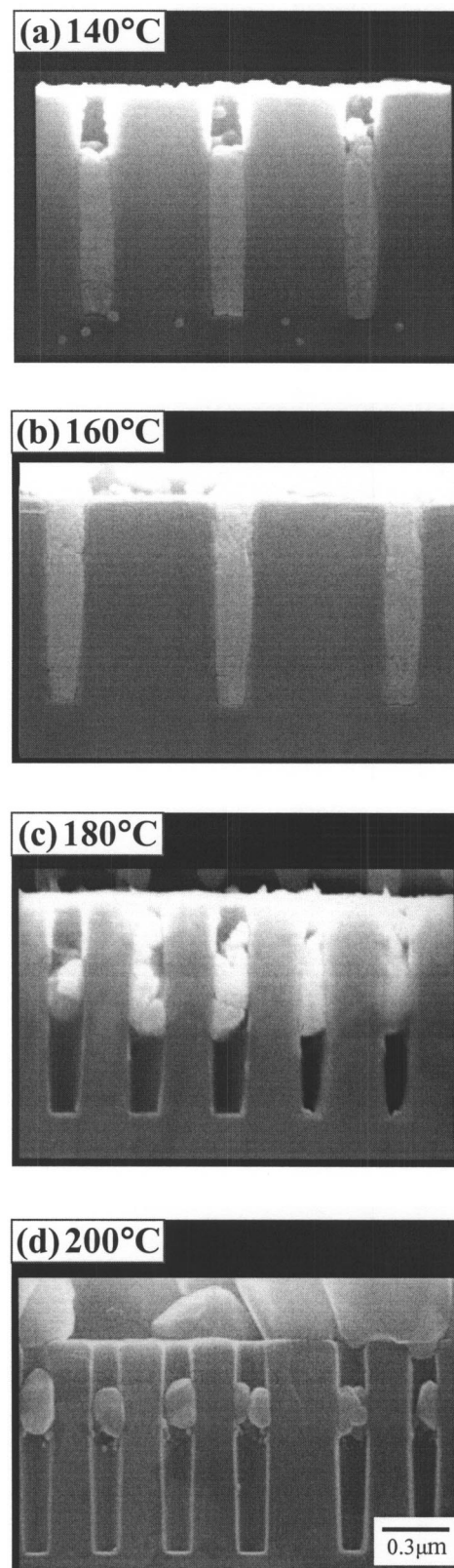


Figure 7. SEM micrographs showing cross-sectional views of via filling for 0.11 μm diameter vias ($\text{AR} = 9.1$) with case B condition at a deposition temperature of (a) 140°C, (b) 160°C, (c) 180°C, and (d) 200°C.

Table III. Results of via filling by CVD Cu film in vias of various sizes at different temperatures.

Processing conditions	Via sizes (μm)	Deposition temperatures ($^{\circ}\text{C}$)				
		120	140	160	180	200
Case A	0.11	F ^a	F	F	F	F
(P = 150 mTorr,	0.15	F	F	F	F	F
He = 25 sccm)	0.18	OK ^b	OK	F	F	F
Case B	0.11	OK	OK	OK	F	F
(P = 60 mTorr,	0.15	OK	OK	OK	F	F
He = 12 sccm)	0.18	OK	OK	OK	F	F

^a F: voids are present in vias.

^b OK: Cu completely filled into vias (void free).

re-emission event) for Cu containing species [Cu(hfac)TMVS or $\text{Cu}^{+1}(\text{hfac})$] within the vias/trenches structure is essential to obtain a good conformality and void-free vias/trenches filling capability.¹⁰ For the deposition conditions at low temperatures (below 160°C) and a high pressure of 150 mTorr (case A), the reactive sticking coefficient S_c of the precursor species is presumably too high such that the Cu containing species can not reach the bottom of the small vias with diam of 0.11 and 0.15 μm . As a result, Cu easily nucleated at the top of a via and on the flat substrate surface, leading to pinch-off of via opening and void formation inside the via.

On the other hand, the deposition conditions of case B is much more favorable for via filling compared with that of case A. This is presumably due to its higher concentration of Cu containing species in the gas phase of CVD (because of the lower flow rate of carrier gas) and the lower deposition pressure. The higher concentration of Cu containing species lead to the higher probability of re-emission and/or desorption event, resulting in a decreased S_c and thus an improved capability of void-free via filling. Moreover, the gas diffusion coefficient (D) is higher at lower total pressure based on the simple gas relation $D \propto T^{3/2}/P_t$, where T is the gas temperature and P_t is total pressure.⁴ Thus, the precursor species was supplied rapidly to the substrate surface at lower pressure and was distributed uniformly under surface-reaction-controlled region. Inside the sub-micrometer vias, the precursor species is supplied by Knudsen diffusion wherein the mean free path of species is longer than the diameter of the via.^{11,26} Thus, the precursor species collide with the walls of via more frequently compared with the collision between the precursor species. Consequently, there is a higher frequency (or probability) of re-emission and/or desorption event within the vias, leading to a decreased S_c and thus a better capability of void-free via filling. However, increasing the deposition temperature would result in the increase of deposition rate of Cu film and the decrease of probability of re-emission and/or desorption event due to enhancement of the probability of $\text{Cu}^{+1}(\text{hfac})$ species decomposition (Eq. 3) at its point of impingement because of the higher thermal energy. As a result, the S_c increased, leading to degraded capability of void-free via filling by CVD Cu film. Therefore, lower deposition temperature is favorable for void-free via filling. In this work, we achieve void-free via filling by CVD Cu film in 0.11 μm diam vias with an AR of 9.1 at and below deposition temperatures of 160°C .

The effects of precursor species concentration in the gas phase and the deposition pressure were investigated with respect to the capability of via filling. Figure 8 illustrates the cross-sectional view SEM micrographs for Cu via-filling at low concentration of precursor species (low precursor flow rate of 0.1 cm^3/min) as well as high deposition pressure (150 mTorr). Since the probability of re-emission event was reduced due to either lowered concentration of precursor species or increased deposition pressure, the reactive sticking coefficient S_c was increased, resulting in the degradation of via-filling capability, as evidenced by comparing the results of Fig. 8 with that of Fig. 7a (deposited with case B condition at 140°C). Moreover, by comparing the results shown in Fig. 8b and Fig. 6a,

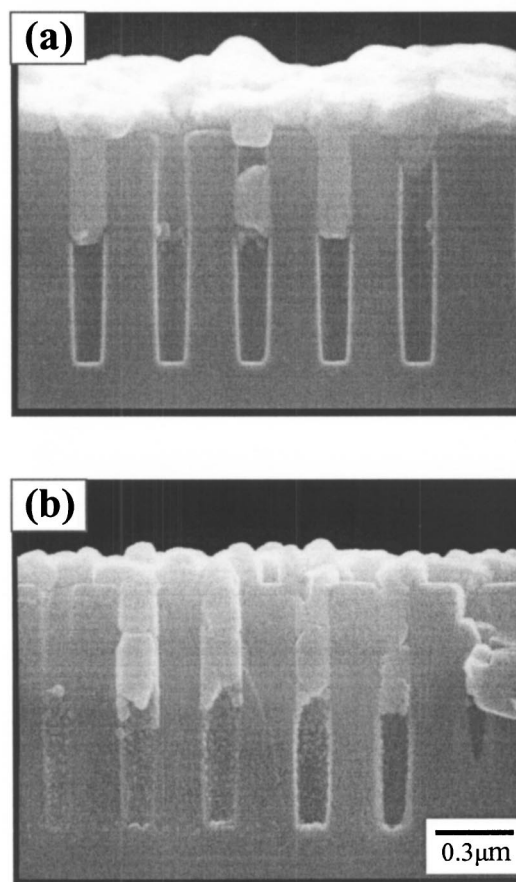


Figure 8. SEM micrographs showing cross-sectional views of via filling for 0.11 μm diameter vias (AR = 9.1) at 140°C with (a) case B condition except reducing the Cu precursor flow rate to 0.1 cm^3/min and (b) case B condition except increasing the deposition pressure to 150 mTorr.

which correspond to the carrier gas flow rates of 12 and 25 sccm, respectively, with other conditions all identical, we found that the lower flow rate of carrier gas (Fig. 8b) resulted in a slightly better via filling because a higher probability of re-emission could be obtained. A superior void-free via filling by Cu film can be achieved at low temperature, low pressure, and high concentration of precursor species in the gas phase; however, these favorable conditions for void-free via filling by Cu film also lead to a slightly degraded film properties, as compared with those obtained under the optimal deposition condition. Apparently, a trade-off is needed between a void-free via filling and a superior film property.

Effects of carrier gas on via-filling.—The characteristics of via-filling are also dependent on the carrier gas used in the CVD of Cu film. In this work, comparison is made between the carrier gas of helium (He) and hydrogen (H_2) using the deposition conditions of case B. Figure 9 shows the cross-sectional view SEM micrographs for Cu via filling in 0.11 μm diameter vias using H_2 as the carrier gas. Void-free via filling by Cu film is achieved at a deposition temperature of 140°C (Fig. 9a); however, voids are present at the bottom of the vias at a deposition temperature of 160°C (Fig. 9b). Table IV shows the comparative results of via-filling by Cu film in 0.11 μm diam vias using different carrier gases of He and H_2 with the deposition conditions of case B. It is found that the maximum deposition temperature of void-free via filling using H_2 as the carrier gas is 140°C , as compared to 160°C using He as the carrier gas.

It was reported that the H_2 carrier gas can enhance the deposition of Cu by the HRR (Eq. 5).¹⁸ In the case of via-filling, the Cu containing species are adsorbed on the wall surface of vias, and then

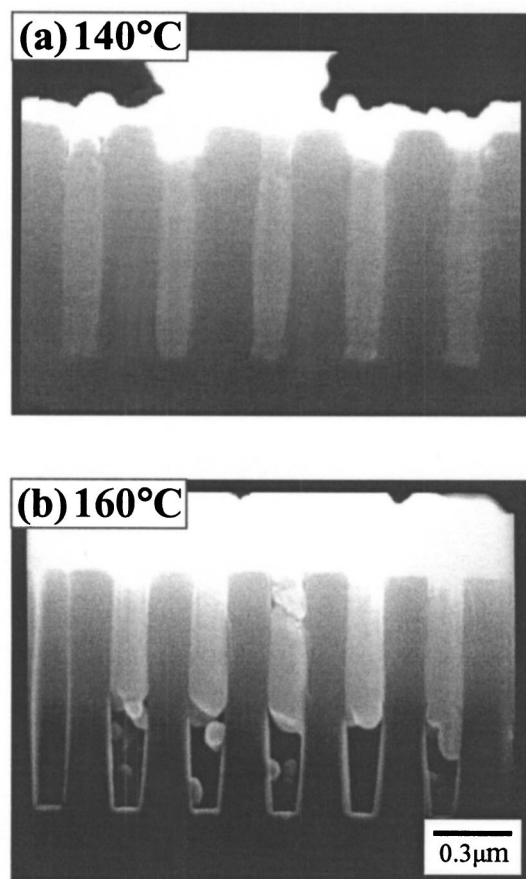


Figure 9. SEM micrographs showing cross-sectional views of via filling for 0.11 μm diameter vias (AR = 9.1) using H_2 as the carrier gas at a deposition temperature of (a) 140°C and (b) 160°C with case B condition.

they can re-emit or nucleate at the points of adsorption by way of the HRR; moreover, the disproportionation reaction (Eq. 3) also proceeds simultaneously. Because of the increasing probability of Cu nucleation, the probability of re-emission and/or desorption event would be reduced, leading to an increased S_c and thus an inferior capability of void-free via filling. From the viewpoint of void-free via filling by Cu film, helium (He) is preferable to hydrogen (H_2) as the carrier gas.

Conclusion

The via-filling properties of CVD Cu film for deep submicrometer vias (diam of 0.11, 0.15 and 0.18 μm) were investigated with respect to various deposition parameters. A superior void-free via filling by Cu film can be achieved at low temperature, low pressure, and high concentration of precursor species in the gas phase of CVD. However, these favorable conditions for void-free via filling

Table IV. Comparison of via filling by Cu film in 0.11 μm diam vias using different carrier gases of He and H_2 with the deposition conditions of Case B.

Carrier gases	Deposition temperatures (°C)				
	120	140	160	180	200
He	OK ^a	OK	OK	F ^b	F
H_2	OK	OK	F	F	F

^a OK: Cu completely filled into vias (void free).

^b F: voids are present in vias.

by Cu film also lead to a slightly degraded film properties, including higher film resistivity, higher content of impurities, degraded adhesion to substrate, and lower deposition rate, as compared with those obtained under the optimal deposition condition. Thus, a trade-off is needed between a void-free via filling and a superior film property. The key to achieve a void-free via filling by Cu film is to have a high probability of re-emission event for the Cu-containing species inside the vias; this is equivalent to have a low reactive sticking coefficient for the Cu-containing species. In this respect, using helium (He) as the carrier gas is preferable to using hydrogen (H_2) because the HRR would enhance the deposition of Cu, resulting in a decreased probability of re-emission event. In this study, we have achieved void-free filling by CVD Cu film in deep submicrometer 0.11 μm diam vias with an AR of 9.1 at and below deposition temperatures of 160°C, using a precursor flow rate of 0.4 cm^3/min and at a deposition pressure of 60 mTorr.

Acknowledgments

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