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유기금속화학기상증착법에 의해 증착된 구리 핵의 기판과 전처리의 의존성

(Substrate and pretreatment dependence of Cu nucleation by metal-organic chemical vapor deposition)

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요 약

Si, SiO_2, TiN, W_2N 기판 위에 (hfac)Cu(VTMS) 유기금속 전구체로 증착된 구리 핵을 조사하였다. 증착온도가 증가함에 따라, 기판 종류에 상관없이 180℃에서 구리 핵이 클러스터링으로 성장하는 메커니즘을 관찰하였다. 또한, HF용액으로 세척한 TiN 과 SiO_2가 공존하는 기판에서 구리 핵의 선택성이 향상됨을 관찰하였다. TiN을 H_2O_2로 세척한 후 Dimethyldichlorosilane 처리했을 때 표면이 passivation됨을 확인하였다.

Abstract

The nucleation of copper(Cu) with (hfac)Cu(VTMS) oganometallic precursor is investigated for Si, SiO_2, TiN, and W_2N substrates. As the deposition temperature is increased, the dominant growth mechanism is observed to change from the nucleation of Cu particles to the clustering of Cu nuclei around 180℃, independent of the employed substrates. It is also observed that the cleaning of substrate surfaces with the diluted HF solution improves the selectivity of Cu nucleation between TiN and SiO_2 substrates. Dimethyldichlorosilane treatment is found to passivate the surface of TiN substrate, contrary to the generally accepted belief, when the TiN substrate is cleaned by H_2O_2 solution before the treatment.

I. INTRODUCTION

As the integrated circuit (IC) technology reaches up to giga-scale integration, the scaling down of aluminum (Al) interconnects lower than 0.25 μm design rule provokes severe problems such as the increase of RC delay time, heat generation and electromigration[1]. Copper (Cu) is the most promising candidate to replace Al in future ICs because of its

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low electrical resistivity and high electro- and stress-migration resistances[2-3]. Despite of its merits, however, Cu has some drawbacks. Cu is a fast diffu ser in Si and SiO_2, and is difficult to remove by wet or dry etching[4]. These drawbacks make Cu difficult to be employed in mass production as a sputtering process or chemical vapor deposition (CVD) process. Fortunately, it has been found that thin film such as titanium nitride (TiN), tungsten nitride (W_2N), or ta ntalum silicon (Ta-Si) amorphous alloys acts as a successful barrier for Cu diffusion[5-7]. On the contrary, the patterning of Cu interconnects by anisotropic etching has been found to be ex tremely difficult[4]. Thus, the selective Cu CVD has been regarded as a potential metalization process in deep submicron IC applications[8].

The selective deposition of Cu films can be achieved with some different processes. For example, the selective Cu CVD can be achieved by ionizing the Cu particles that are going to be deposited for a specified regions[9]. One can also selectively deposit Cu films on the metallic surface compared to the dielectric one when a metal pattern is already formed on the dielectric substrate. This type of selective Cu CVD is due to the enhanced nucleation at the metallic surface because of the easy charge transfer from the metal surface needed in the disproportionation reaction described in eq. (1)[8]. The selective Cu CVD on the metallic surface is su itable for th e Cu interconnects In ICs, since the metallic film used for the enhanced Cu deposition can be employed simultaneously as a diffusion barrier. But, this selective Cu CVD on the metallic surface can be affec ted by the substrate temperature, deposition rate and the existence of impurity atoms at the surface, as well as the properties of substrate[8, 10]. The selectivity window is known to decrease as the deposition temperature and/or depositi on rate are increased[8]. The hydroxyl groups (-OH) act as chemisorption sites for (hfac)Cu(I) precursors at the surface[11-12]. Thus they are typical impurities that affect the selectivity of Cu CVD. Although the intentional modification of surfaces is known to affect the selectivity of Cu CVD[11], a number of contradictory results still persist[11]. This fact reflects that different surface treatments result in different nucleation behavior of Cu particles. Therefore, a detailed study on the nucleation behavior in Cu CVD, for the combinations of metallic and dielectric surfaces and under various surface treatment conditions, would be important to clarify the observed controversy.

In this paper, we have studied the deposition behaviors of Cu CVD on native oxide-covered Si, thermally-grown SiO_2, titanium nitride (TiN), and tungsten nitride (W_2N) substrates. We have also studied the selectivity of Cu CVD on the TiN/SiO_2 surfaces after various chemical treatments.

Ⅱ. EXPER IMENTAL DETAILS

The substrates employed in this study we re boron-doped p-type (100) Si wafer, 3000 Å-thick SiO_2 layer grown on (100) Si wafer by wet oxidation at 1000℃, about 1500 Å-thick TiN and W_2N films grown on thermal SiO_2 layers. The TiN films, deposited by reactive sputtering from the Ti target in N_2 ambient, had a polycrystalline columnar structure with <111> growth direction, The W_2N films were deposited by plasma-enhanced CVD (PECVD) using ammonia (NH_3) and tungsten hexafluoride (WF_6) gases, and had an amorphous structure.

The Cu films were deposited by metal-organic CVD (MOCVD) using the hexafluoro-acetylacetonate copper vinyltrimethylsilane, (hfac)Cu(VTMS), a ligand stabilized Cu(I) β-diketonate. We employed this precursor since its liquid phase at room temperature enables easy handle and the VTMS li gand is weakly bound to the Cu in the precursor molecule, which leads to the prompt decomposition of the precursor at relatively low temperatures (about 150~200℃). In the Cu deposition process, the precursor is known to undergo a reversible

disproportionation reaction[13-14],

$$2(hfac)Cu(VTMS)_{[g]} \rightarrow Cu_{[s]} + Cu(hfac)_{2[g]} + 2VTMS_{[g]} \quad (1)$$

to form solid copper. The MOCVD was performed at 0.5 Torr with helium (He) carrier gas. The substrate temperature was varied between 150℃ and 220℃. The structural properties of deposited Cu films were monitored by scanning electron microscope (SEM).

The influence of chemical treatments on the nucleation behavior and the selectivity of Cu deposition were studied for various surface cleaning and/or passivation processes. All the substrates were treated by the normal cleaning process using trichloroethylence (TCE), aceton, methanol, deionized (DI) water, and then dried by N_2 gas blow. Some Patterned substrates were dipped in the SC1 cleaning solution (NH_4OH : H_2O_2 : DI water = 1 : 1 : 5) or the diluted hydrofluoric acid (HF : DI water = 1 : 10) for 15 sec before DI water cleaning and drying. The surface of some other patterned samples were passivated by dimethyldichlorosilane(DMDCS : $(CH_3)_2SiCl_2$). In this case, the sam ple surface was cleaned in the boiling 4% H_2O_2 solution before the passivation to eliminate the organic and inorganic surface conta mination and then followed by the DI water cleaning and drying. The surface passivation by DMDCS was performed by dipping the samples in the boiling DMDCS for 3 min followed by N_2 gas blowing.

III. RESULTS AND DISCUSSIONS

The nucleation behavior is studied for two kinds of dielectric materials (Si and SiO_2) and two kinds of diffusion barrier metals (TiN and W2N) to get an idea for the appropriate metal/dielectric combination for the selective Cu CVD. Figures 1 and 2 show the temperature dependence of surface morphology when the MOCVD is perfo rmed for 1 min on the normally cleaned Si and SiO_2 substrates, respectively. We can easily compare the initial stages of Cu nucleation on Si and SiO_2 substrates in these figures. Cu nucleation is observable when the substrate temperature is higher than 180℃ in the case of Si (Fig. 1), but it is not observable until 180℃ on the SiO2 substrate (Fig. 2). This means that the Cu nucleation is easier on the Si surface compared to surface of SiO_2. Since the conductivity of Si substrate is much higher than that of SiO_2, the easy Cu nucleation on Si substrate can be easily explained from the easy electron transfer from the substrate needed in the Cu nucleation[14]. The charge transfer from the substrate to the adsor bed species would be governed by the two factors, the electron affinity of the dielectrics (work function in the case of metal substrate) that determines the easiness of electron transfer and the conductivity of the dielectrics that is related to the amount of available electrons. The electron affini ty of SiO_2 is much smaller than that of Si which has the value of 4.05 eV. Therefore, SiO_2 should be more catalytically active than Si when enough electrons are existent in the SiO_2 conduction band, since the electron transfer to the adsorbed species from the SiO_2 substrate should occur more easily due to its lower electron affinity. Thus, the ob served fact means that the conductivity of the substrate determines the electron transfer from the substrates in this case. It should be noted, however, that our Si substrate is p-type in which electrons are minority carriers and the surface of our Si substrate is covered with the native oxide after our normal surface cleaning process. Therefore, the easy nucleation on the Si substrate means that the free electrons are transferred from Si substrate to the organic molecules though the quantum tunneling across thin native oxide. The results in Fig. 1 and 2 also mean that the metal/SiO_2 pattern would be more effective in the selective Cu deposition than the metal/Si pattern.

3 shows the dependence of the average Cu nuclei density and the average grain size on the deposition temperature when the Cu CVD is performed on Si and SiO_2 substrates for one min. For the measure

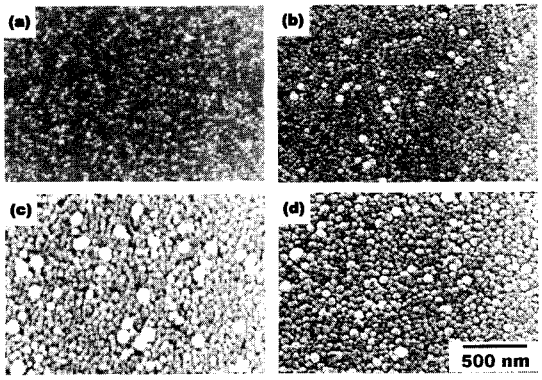

그림 1. Si 기판 위에 1분간 증착된 구리 입자의 SEM 사진 (a) 150℃, (b) 180℃, (c) 200℃, (d) 220℃

Fig. 1. SEM micrographs of Cu particles deposited on the (100) p-type Si substrate for one min at 150℃(a), 180℃(b), 200℃(c), and 220℃(d).

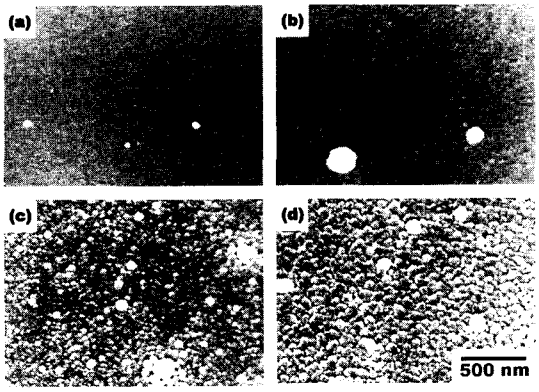

그림 2. SiO_2 기판 위에 1 분간 증착된 구리 입자의 SEM 사진 (a) 150℃, (b) 180℃, (c) 200℃, (d) 220℃

Fig. 2. SEM micrographs of Cu particles deposited on the SiO_2 substrate for one min at 150℃ (a), 180℃(b), 200℃(c), and 220℃(d).

ments, the area of 2×2 cm^2, where the nucleus density is highest(near the center of the wafer), was analyzed by the SEM observations. One can easily see, in this figure, that the average grain size on Si substrate is larger than that on SiO_2 substrate under the same deposition temperature. One can also notice that nuclei density increases sharply with the deposition temperature up to 180℃, and then decreases sharply as the temperature is increased further. The former phenomenon below 180℃ is evidently related to the increased nucleation reaction of the substrate due to the increase of precursor dissociation at higher temperatures. Close observation of the SEM micrographs in Figs. 1 and 2, show that the higher the deposition temperature, the greater portion of the surface of Si and SiO_2 substrates is covered by the Cu film. Therefore, the decrease of nuclei density above 180℃ in Fig. 3 suggests that the coalition of Cu nuclei into clusters begins at 180℃ even though further nucleation can not be ignored above this temperature. Actually, we believe that the coalition of Cu nuclei begins even below 180 ℃ from the fact that the nucleus density on SiO_2(with small grain sizes) is larger than that on Si(with large grain size) for the samples deposited at 180℃. Anyway, it is evident that Cu film grows on Si and SiO_2 substrate with the island type, i.e., with the Volmer-Weber growth mechanism. The fact that the resistivity of Cu film increases when the Cu CVD is performed at the temperatures above a certain critical temperature(220℃ in the case of ref.

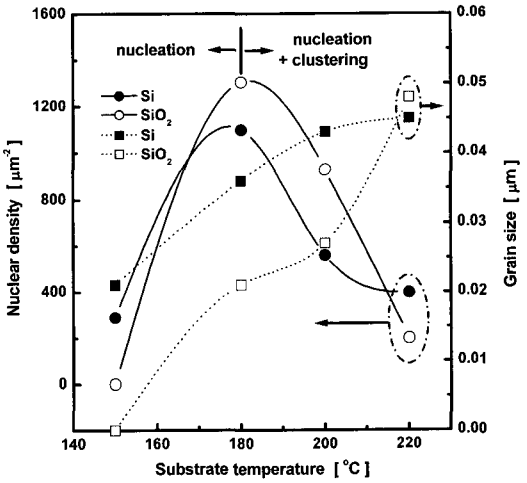

그림 3. Si와 SiO_2 기판 위에 1분간 증착된 구리 박막의 증착온도에 따른 핵 밀도와 그레인 크기

Fig. 3. Deposition temperature dependences of average nucleus density and grain size of Cu films deposited on Si and SiO_2 substrates for one min. The solid and dotted lines are for the guide of view. Note the nucleus density decrease above 200℃ due to the clustering of Cu nuclei.

15) should be related to this growth mechanism. That is, more voids would be formed between the clus ter islands when the cluster formation is too rapid under high deposition rate.

The nucleation behavior of Cu on TiN and W_2N substrates are shown in Fig. 4 and Fig. 5, respec-tively. One can notice in these figures that the Cu nucleation is easier on W_2N than on TiN substrate. To find out the physical mechanism that provokes this difference, we examined the surfaces of these substrates using optical microscope and measured the resistivity of the substrates. We observed that the surf ace of amorphous W_2N substrate is smoother than that of polycrystalline TiN substrate, and that W_2N substrates is more conductive than the TiN one, even though the former substrates show rather scattered resistivity. The kinetics of surface chemical reactions is necessarily affected by the binding site preferences. It is generally believed that the defects, and steps in particular, are the locales where the reactivity is maximum. Therefore, for the nucleation of W_2N substrate to be faster than that on TiN substrate, it is evident that there should be some mechanisms that overcome the enhanced reactivity at the steps. One reason may be higher conductivity of W_2N substrate. That is, the electron transfer to the precursor needed in Cu nucleation may be easier on more conductive W_2N substrate. We believe, how-ever, that the hydrogen atoms resting at the surface of our W_2N substrate would be the main origin of the enhanced surface reactivity of W_2N substrate, as will be discussed in the next paragraph. We can also confirm that copper nucleation is easier on the metallic surface (Fig. 4, 5) than on the surface of semiconductor or insulator (Fig. 1, 2). This pheno-menon should be related to the difference in the charge transfer from the metallic substrate and that fro m the dielectric substrate.

Figure 6 shows the dependence of average nucleus density and grain size on the deposition temperature when Cu CVD is performed on TiN and W_2N substrates for one min. One can notice again in this

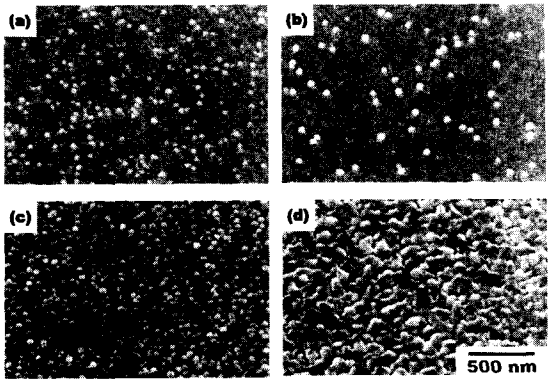

그림 4. TiN 기판 위에 1분간 증착된 구리 입자의 SEM 사진 (a) 150℃, (b) 180℃, (c) 200℃, (d) 220℃

Fig. 4. SEM micrographs of Cu films deposited on the TiN substrate for one min at 150℃(a), 180℃(b), 200℃(c), and 220℃(d). Polycrys-talline TiN substrate provided by sputter deposition on Si substrate was employed for this study.

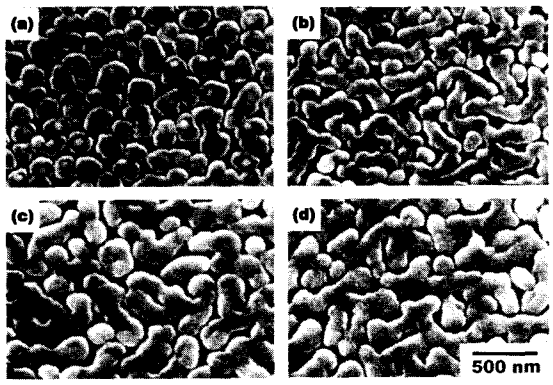

그림 5. W_2N 기판 위에 1 분간 증착된 구리 입자의 SEM 사진 (a) 150℃, (b) 180℃, (c) 200℃, (d) 220℃

Fig. 5. SEM micrographs of Cu films deposited on the W_2N substrate for one min at 150℃(a), 180℃(b), 200℃(c), and 220℃(d). The W_2N substrate was provided by PECVD and had amorphous structure.

figure that the Cu film on W_2N substrate has higher nuclei density and larger grain size than that on TiN substrate. As mentioned in the previous paragraph, this phenomenon may be related to the difference of substrate c onductivity but not to the surface roughness of the substrate. Even if the higher conductivity of W_2N substrate is taken into account,

however, the Cu film on W_2N substrate seems to have much denser nucleus density and much larger grain size compared to that on TiN substrate. For example, the Cu film on W_2N substrate grown at T_d = 180℃ has about three times higher nucleus density and ten times larger grain size than that on TiN substrate. We believe, therefore, that this great difference is related to the existence of hydrogen atoms in our W_2N substrate provided by PECVD using NH_3 and WF_6 gases. According to Awaya and Arita, Cu deposition rate is greatly enhanced when H_2 is used with (hfac)Cu(VTMS) as the carrier gas[16]. They have observed also that the Cu films provided with H_2 carrier gas show similar resistivity and optical reflectivity with the one provide d with Ar gas when the deposition rate is rather slow under 200℃. But the resistivity (reflectivity) of the former films becomes much smaller (larger) than those of the latter films. To explain all these phenomena, they proposed that H_2 carrier gas induces more nucleation. Actually, the $Cu(hfac)_2$ gases which are by-products in eq. (1) can be decomposed into Cu via a hydro-genreduction reaction,

$$Cu(hfac)_{2[g]} + H_{2[g]} \rightarrow Cu_{[s]} + 2H(hfac)_{[g]} \qquad (2)$$

in parallel with a disproportionation reaction of eq. (1). In our case, in which inert He gas is used as carriers, the hydrogen impurity atoms at the W_2N surface will enhance Cu nucleation. Then a higher deposition rate will continue after the initial enhancement of Cu nucleation, since the deposition rate of copper on Cu nuclei is faster than that on TiN as obs erved in the island-type growth.

In Fig. 6, we observed once again that the grain size (nucleus density) of the film deposited above 180℃ increases (decreases) with the temperature due to the dominance of clustering phenomena. This result and the result i n Fig. 3 suggest that growth mechanism changes from main nucleation to the nucleation of Cu particles and coalition of grains around 180℃ independent of substrate. To verify whether this temperature is independent of the deposition rate, the precursor flow rate is increased or reduced twice with the fixed deposition time of one min except for the case of SiO_2 substrate(where deposition time is increased twice when the flow rate is reduced twice to see the size and density of Cu particles more cleanly). We observed that the change of growth pattern begins around 180℃, independent of the substrate even when the precursor flow rate is changed. That is, the variation of flow rate induced only average nucleus density and grain sizes showing nearly identical temperature dependence of growth pattern. This result is quite a nomalous, since some activation energy is required for the chemisorbed Cu particles to migrate to form clusters and this activation energy should depend on the nature of surface as widely known in epitaxial growth experiments. One possible reason might be sought from the fast diffusion rate of Cu particles. It is well known that Cu is a fast diffuser in nearly all semiconductors and dielectric materials. Actually such a value as low as 0.28 eV is reported for the activation energy of the self-diffusion of Cu adatoms on Cu (100) surface[17]. Moreover, the Cu dimmers contribute as much to the self-diffusion as Cu adatoms[18]. The fact that the dissociation barrier for a dimmer is much larger than the activation energy of self-diffusion on the surface suggests also that the Cu particles migrate on the Cu (100) surface in the form of clusters rather than the individual atom[17 18]. If Cu diffuses very fast at the surface of the employed substrates with small activation energy forming clusters, the dependence of activation energy on the nature of substrate surface would be difficult to observe and the temperature where the change of growth pattern begins would not be scattered for the employed substrates.

A wide processing window is essential for the selective Cu CVD to be useful in actual fabrication. Moreover, a continuous planar type Cu film with low res istivity should be grown on the metal substrate for the metal layer to be used as a diffusion barrier under the Cu interconnects. Our results up to now

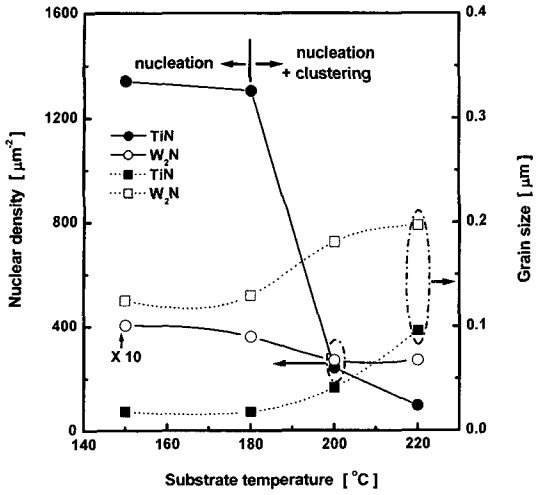

그림 6. 그림6.TiN과 W_2N 기판 위에 1분간 증착된 구리 박막의 증착온도에 따른 핵 밀도와 그레인 크기

Fig. 6. Deposition temperature dependences of average nucleus density and grain size of Cu films deposited on TiN and W_2N substrates for one min. Note nucleus density(grain size) decrease(increase) sharply above 180℃ (d) due to the clustering of Cu nuclei.

indicate that a combination of W_2N and SiO_2 surfaces would be the most appropriate for selective Cu deposition in our four possible surface combinations. We thus studied the dependence of selectivity between TiN and SiO_2 surfaces on the chemical treatment conditions, since a good selective deposition with a wider processing window can be automatically obtain ed with W_2N/SiO_2 surfaces when we can get a good enough selective deposition conditions between TiN and SiO_2 surface. The Cu CVD proceeds with Cu nucleation, followed by grain growth as observed in the above results. Successful selective Cu deposition thus depends on the appropriate control of Cu nucleation. In this context, we studied the influence of cleaning process on the nucleation behavior.

Figure 7 shows typical SEM micrographs obtained when the Cu deposition is performed at 150℃ for 5 min. We can easily notice that the surface treatments affect the selectivity of Cu deposition. When the patterned surface is treated by a standard cleaning process [Fig. 7(a)], the Cu film is grown continuously on the TiN surface, and some Cu grain s are nucleated on the SiO_2 surface. When the sample is cleaned by a diluted HF solution [Fig. 7(b)], the patterned surface shows a perfect selectivity. In this case, however, the Cu film grown on TiN surface is rather of granular form than being continuou s. This fact means that the trace of HF treatments remnant at the surface impedes the Cu nucleation regardless of the nature of substrate. We can observe in Fig. 7(c) and 7(d) that the selective deposition is completely destroyed after the SC1 cleaning or DMDCS treatments. We can also notice that the SC1 cleaning impedes most effectively the nucleation of copper among the studied surface treatment conditions. The SC1 cleaning is known to leave thin native oxide layer after the etching of organic and/ or in o rganic contaminants. We thus believe that this newly-formed native oxide is very resistant to the copper nucleation. On the other hand,

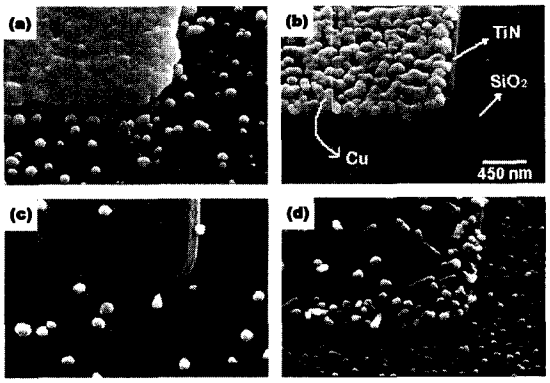

그림 7. 패터닝된 TiN/SiO_2 기판 위에 150℃에서 5분간 증착된 구리 박막의 SEM 사진 (a) 일반 세척 공정, (b) HF 세척 공정, (c) SC1 세척 공정, (d) DMDCS 처리

Fig. 7. SEM micrographs of Cu films deposited on the patterned TiN/SiO_2 substrate for five min at 150℃. The substrate surfaces are treated with (a) normal cleaning chemicals, (b) diluted HF solution, (c) SC1 cleaning solution, and (d) DMDCS solution for 3 min just before Cu deposition. Note the diluted HF solution treatments give nearly perfect selectivity under the employed deposition conditions.

DMDCS is known to passivate the surface OH radicals which enhance the copper nucleation[11]. Thus the in-situ injectio n of DMDCS inhibits the nucleation of copper on the PECVD oxides[19], while it does not affect the Cu nucleation on the metallic surface since DMDCS does not bond with the metal surface[20]. Actually, Webb et al. observed that the growth rate of Cu film increased slightly with DMDCS passivation, even though they could not explain the chemical reason of the increased growth rate. However, our results in Fig. 7 show clearly that the Cu nucleation on the metallic surface is also greatly reduced after our DMD CS passsivation. To understand the mechanism of this phenomenon, we observed the nucleation behavior after a simple dipping of the sample in the boiling.

Ⅳ. CONCLUSION

We have investigated the nucleation of Cu particles on Si, SiO_2, TiN, and W_2N s ubstrates in the MOCVD of Cu film using (hfac)Cu(VTMS) precursor. It is found that the dominant film growth mechanism changes from the nucleation of Cu particle at low deposition temperatures to the coalition of deposited Cu nuclei around 180℃, independent of the employed substrates. The W_2N substrate provide d by PECVD is observed to be more effective than the sputter-deposited TiN substrate for the selective deposition of Cu film. The cleaning of substrate surfaces with diluted HF solution and/or SC1 clean ing solution is observed to impede the nucleation of Cu particles on the metallic substrate as well as the dielectric substrate. We have also observed that the selectivity of Cu deposition between TiN and SiO_2 substrates is greatly improved after the surface treatments with diluted HF solution. The DMDCS treatment, which is usually known to be noninteractive with the metallic surface, is found to passivate the surface of TiN substrate, contrary to the generally accepted belief.

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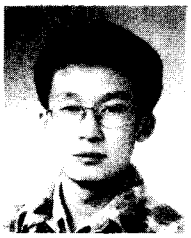

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