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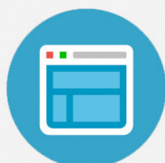

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Effects of the underlayer substrates on copper chemical vapor deposition

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Copper chemical vapor deposition (Cu CVD) on different substrates, including TiN, Ta, and TaN, was studied with regard to the physical property, nucleation, and adhesion of the deposited Cu films. The Cu film deposited on TiN substrate has a number of favorable properties over the films deposited on Ta and TaN substrates: lower electrical resistivity, lower impurity contamination, and higher (111)-preferred orientation. Moreover, CVD of Cu films on TiN substrate has a shorter incubation time and better film adhesion. From the viewpoint of adhesion, microstructure, and physical properties of the Cu film, we may conclude that TiN is a better underlayer substrate than Ta and TaN for Cu CVD. © *2002 American Vacuum Society.* [DOI: 10.1116/1.1481863]

I. INTRODUCTION

Copper has been regarded as a potential candidate to replace Al and its alloys in integrated circuits as the multilevel interconnect material because of its low bulk resistivity (1.67 $\mu\Omega$ cm), excellent electromigration resistance, and high resistance to stress-induced voids.[1,2] To implement copper into metal lines and vias interconnect, the damascene process is developed to cope with the difficult Cu dry-etching problem. However, Cu diffuses rapidly in a Si substrate and forms Cu–Si compounds at low temperatures (about 200 °C), causing deep-level traps in Si. Moreover, Cu adheres poorly to dielectric layers and drifts through the oxide under field acceleration.[3–5] Therefore, a diffusion/adhesion barrier between Cu and its surrounding layer is necessary for successful application of Cu in silicon-based integrated circuits. There are various techniques of copper film deposition, such as chemical vapor deposition (CVD),[6,7] conventional and ionized metal plasma (IMP), physical vapor deposition,[8] and electrochemical deposition (ECD), including electroplating and electroless plating;[9,10] among these, the CVD method has the advantages of superior step coverage and excellent gap-filling capability for high-aspect-ratio vias and trenches,[7] making it a promising technique of Cu film deposition for future integrated-circuit (IC) applications. Although the Cu ECD combined with IMP depositions of thin Cu seed layer and barrier layers provide a suitable solution for IC technologies above 0.25 μm, the technique of Cu CVD is favorable 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 subquarter-micron vias.[11]

The properties of CVD Cu films, such as the growth rate, film texture, surface morphology, impurity contamination, and adhesion to the underlying layer (barrier), are closely related to the initial nucleation of Cu on the substrates.[12–15] Variation of nucleation and growth mechanisms of CVD Cu on different substrates leads to different properties of the deposited Cu films. A number of barrier materials have been used as substrates for chemical vapor deposition of Cu films, such as W, TiW, TiN, Ta, and TaN;[6,12,14–16] among them, TiN, Ta, and TaN exhibit superior barrier properties against Cu diffusion. In this study, we investigate the effect of different substrates (TiN, Ta, and TaN) on the nucleation and film properties of CVD Cu using a low-pressure CVD system built by ourselves for this purpose. Adhesion of the Cu films is also investigated.

II. EXPERIMENT

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 is a shower-head injector through which the Cu precursor is introduced into the reaction chamber in a stream of carrier gas. Under the injector, there is a substrate susceptor, which can be heated resistively 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 and the precursor injector are 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.[17,18] The 2.4 wt % TMVS enhances the stability of the precursor.[18] 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 pushed by the N_2 gas through the LFC. It is then vaporized in the CEM and mixed with the carrier gas. Helium (He) is used as the carrier gas in this study. The precursor-saturated

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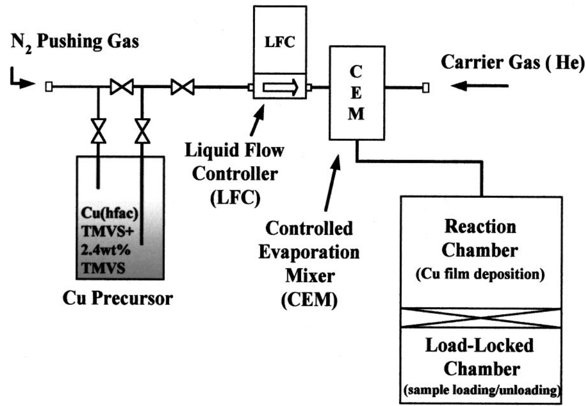


FIG. 1. Schematic diagram of low-pressure Cu CVD apparatus used in this study.

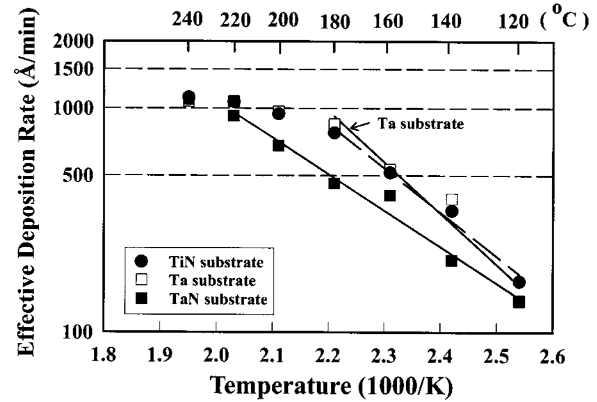


FIG. 2. Deposition rate of Cu film vs substrate temperature (Arrhenius plot) at a pressure of 150 mTorr.

carrier gas is introduced into the reaction chamber through the gas injector for the CVD of Cu films.

In this study, TiN, Ta, and TaN were used as the substrates for CVD of Cu films. All of the substrate layers were 50 nm thick, and were sputter-deposited on Si wafers of 4-in.-diam covered with a thermal oxide (500 nm thickness). A dc magnetron sputtering system with a base pressure of 1.5×10^{-6} Torr was used to reactively sputter-deposit the TiN, Ta, and TaN substrate layers at a pressure of 7.6 mTorr. The TiN substrate was sputter-deposited using a Ti target (99.99% purity) in an Ar/N_2 gas mixture; the flow rates of Ar and N_2 were 60 and 1.5 sccm, respectively. The Ta and TaN substrates were sputter-deposited using a Ta target (99.99% purity) in a pure Ar gas and an Ar/N_2 gas mixture, respectively; the flow rate of Ar was 24 sccm for the Ta film deposition, and the flow rates of Ar and N_2 were 24 and 6 sccm, respectively, for the TaN film deposition.

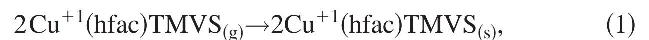
The substrate wafer (coated with TiN, Ta, or TaN) was loaded into the Cu CVD system. When the pressure of the load-locked chamber reached 10^{-6} Torr, the substrate wafer (together with the substrate holder) was transferred to the reaction chamber for Cu film deposition. Prior to starting the Cu film deposition, the substrate sample was heated to the desired deposition temperature with the He carrier gas flowing at 25 sccm and the chamber pressure maintained at 150 mTorr. About 1 h was typically required for the substrate sample to reach the preset temperature. In this study, Cu CVD was performed over a temperature range of 120 to 240 °C at a pressure of 150 mTorr with a precursor flow rate of 0.4 mL/min and a He carrier gas flow rate of 25 sccm. At the end of Cu film deposition, the sample was cooled in ambient He at a pressure of 150 mTorr.

The thickness of Cu films was measured by a DekTek profiler on the patterned Cu films and was verified by cross-sectional scanning electron microscope (SEM) examination. SEM was also used to observe the surface morphology of the deposited Cu films. A four-point probe was employed to measure the sheet resistance. Secondary-ion mass spectrometry (SIMS) was used to analyze the impurity content in the Cu films. Crystal structure was identified by x-ray diffraction (XRD) analysis. The surface roughness of Cu and various substrate layers was evaluated by atomic force microscopy (AFM). Rutherford backscattering spectroscopy (RBS) was used to determine the composition of the substrate layers. The Scotch tape pulling test was used to assess the adhesion between the CVD Cu films and the substrate layers.

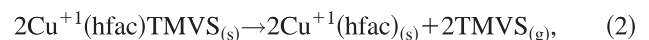
III. RESULTS AND DISCUSSION

A. Film properties of CVD Cu films on various substrates

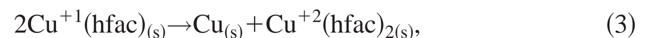
The reactively sputtered TiN, Ta, and TaN substrates have resistivities of 8.50, 0.19, and 0.60 mΩ cm, respectively, and the compositions of the metal nitrides are $TiN_{1.1}$ and $TaN_{1.2}$, as determined by RBS. Figure 2 shows the effective deposition rate of Cu films on different substrates as a function of substrate temperature (Arrhenius plot) at a deposition pressure of 150 mTorr with a He carrier gas flow rate of 25 sccm and a liquid Cu precursor flow rate of 0.4 mL/min. The effective deposition rate of a Cu film was calculated using the thickness deposited in a period of 10 min. The chemical reaction of Cu CVD using Cu(hfac)TMVS as a precursor proceeds on the substrate surface by a facile disproportionation as follows:[19]



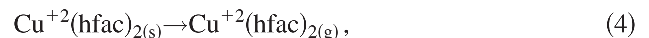
$$2Cu^{+1}(hfac)TMVS_{(g)}\rightarrow 2Cu^{+1}(hfac)TMVS_{(s)}, \quad (1)$$



$$2Cu^{+1}(hfac)TMVS_{(s)}\rightarrow 2Cu^{+1}(hfac)_{(s)}+2TMVS_{(g)}, \quad (2)$$



$$2Cu^{+1}(hfac)_{(s)}\rightarrow Cu_{(s)}+Cu^{+2}(hfac)_{2(s)}, \quad (3)$$



$$Cu^{+2}(hfac)_{2(s)}\rightarrow Cu^{+2}(hfac)_{2(g)}, \quad (4)$$

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^{+1}(hfac) and the substrate surface. Thus, it is easier to deposit Cu films on a conducting substrate than on an insulating substrate. Because the chemical reaction of Cu CVD

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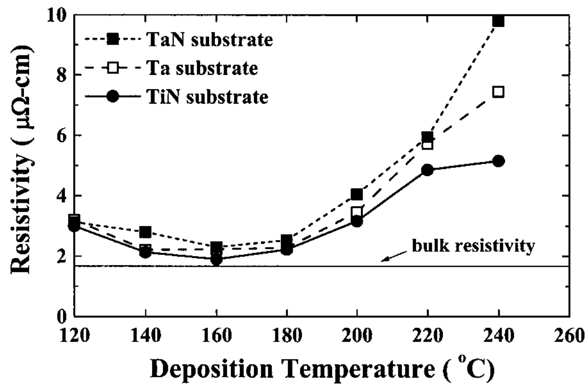

FIG. 3. Resistivity of Cu film vs deposition temperature for Cu films deposited at a pressure of 150 mTorr.

involves a thermal dissociation of the Cu precursor [Eq. (2)], a higher temperature would result in a higher rate of deposition. The activation energy Ea was determined to be 9.04, 10.50, and 7.35 kcal/mole for TiN, Ta, and TaN substrates, respectively, by the Arrhenius equation:

$$R = R_0 \exp(-Ea/kT), \qquad (5)$$

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. The Cu films deposited on TiN and Ta substrates have a higher effective deposition rate than those deposited on the TaN substrate in the surface-reaction-controlled region; however, in the mass-flow-controlled region, Cu films on different substrates have nearly the same effective deposition rate. The resistivity of the Cu film was calculated using the measured sheet resistance and thickness. Figure 3 illustrates the resistivity of Cu films as a function of deposition temperature, at a pressure of 150 mTorr. The resistivity 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, whereas 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 optimal temperature for Cu film deposition for minimization of resistivity under the present deposition conditions appears to be around 160 °C, irrespective of the substrate. Among the three substrate layers used in this study, Cu films deposited on TiN substrates revealed the lowest resistivity over the entire temperature range of 120 to 240 °C, and the resistivity of the film deposited at 160 °C was determined to be 1.9 $\mu\Omega$ cm. Figure 4 illustrates the SIMS depth profiles of Cu films deposited on TiN, Ta, and TaN substrates at a pressure of 150 mTorr. The contamination (impurities) of Cu films is associated with the disproportionation of Cu(hfac)TMVS precursor. It has been reported that TMVS is easily exhausted and not a contributor of contamination at high vapor pressures, however, the hfac molecule is the source of observed impurities of fluorine (F), carbon (C), hydrogen (H), and oxygen (O).[19,21,22] The content of impurities in the Cu film deposited at 220 °C is higher than that in the film deposited at 160 °C [Figs. 4(a) and 4(b)]; this condition results in higher resistivity for the Cu film deposited at 220 °C (Fig. 3). At the same deposition temperature of 160 °C, on the other hand, the content of impurities in the Cu film deposited on TiN substrate is lower than those in the Cu films deposited on the Ta and TaN substrates [Figs.

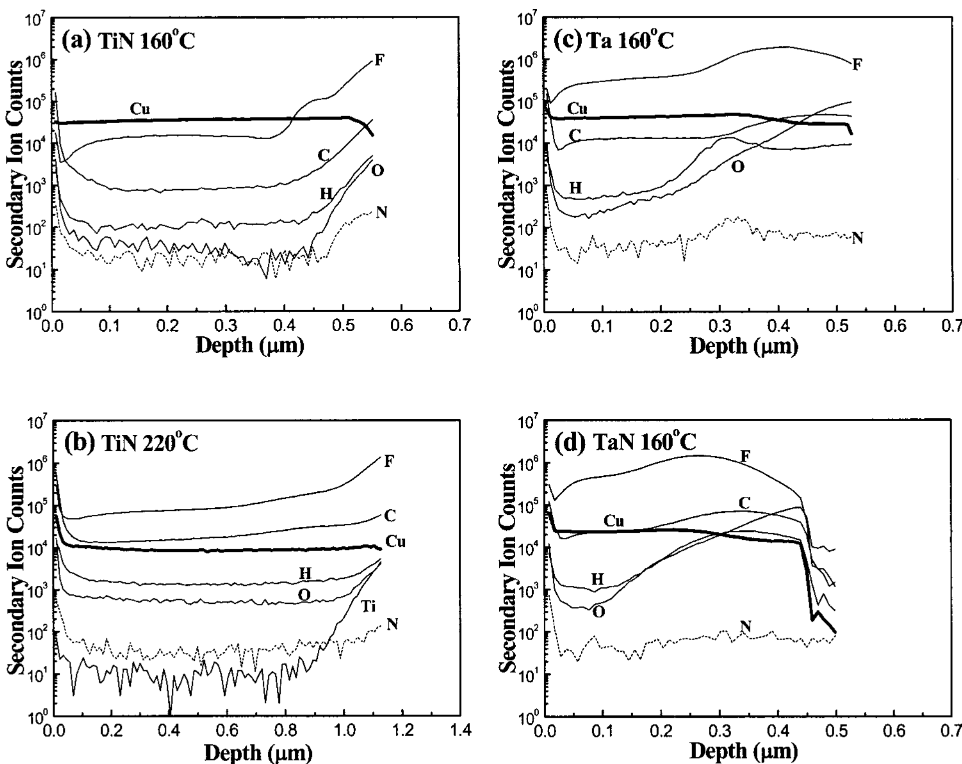

FIG. 4. SIMS depth profiles of Cu films deposited on (a) TiN substrate at 160 °C, (b) TiN substrate at 220 °C, (c) Ta substrate at 160 °C, and (d) TaN substrate at 160 °C. The deposition time is 10 min.

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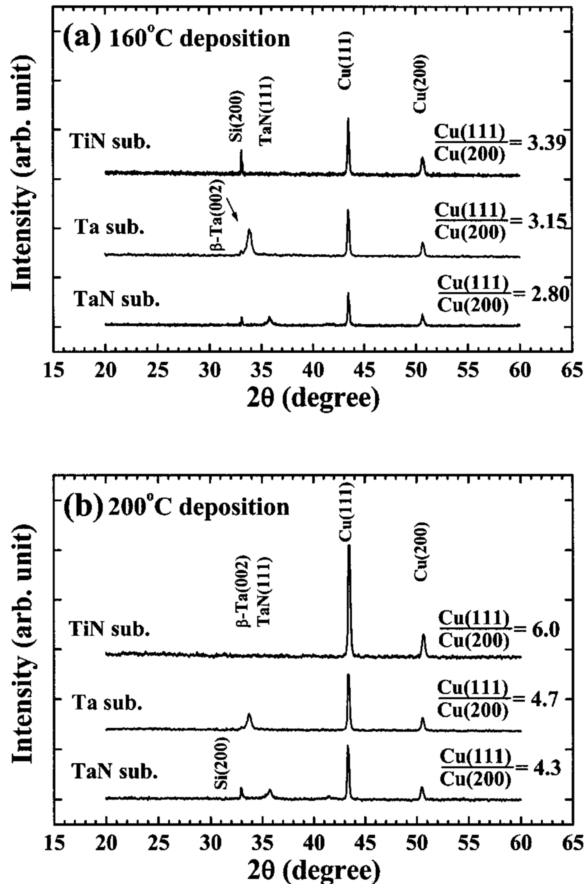

FIG. 5. XRD spectra of Cu films deposited on various substrates at (a) 160 °C and (b) 200 °C.

4(a), 4(c), and 4(d)]; this observation of impurity content is also consistent with the measured film resistivity (Fig. 3). Notably, the impurities at the Cu/substrate interface have a higher content and broader distribution for the Cu films deposited on Ta and TaN substrates compared to that deposited on the TiN substrate. Moreover, it is found that the impurities easily accumulated at the substrate surface in the initial stage of CVD Cu film deposition, especially for fluorine impurities, similar to the report in literature.[21]

Different underlayer substrates would result in different preferred orientation of the deposited Cu films. Figure 5 shows the XRD spectra for Cu films deposited on various substrates. For the Cu film deposited at 160 °C, the intensity peak ratio of Cu(111) to Cu(200) reflections on the TiN substrate was determined to be 3.39, which is higher than the peak ratios observed on the Cu films deposited on Ta and TaN substrates [Fig. 5(a)]. At a higher deposition temperature of 200 °C, all of the peak ratios increased, especially for the Cu film deposited on the TiN substrate [Fig. 5(b)]. Figure 6 illustrates the XRD intensity peak ratio of Cu(111) to Cu(200) reflections vs deposition temperature of Cu films deposited on the three different substrates. Copper films deposited on the TiN substrate revealed the highest peak ratio among the three substrates investigated in this study.

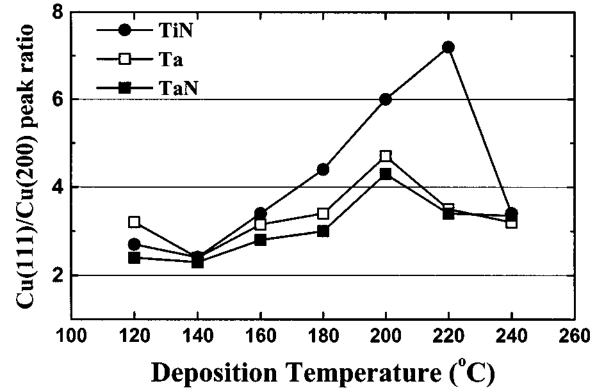

FIG. 6. XRD peak ratio of Cu(111) to Cu(200) reflections vs deposition temperature of Cu films deposited on various substrates.

B. Nucleation and surface morphology

The surface condition of substrates plays an important role in the Cu CVD. Figure 7 shows the AFM images of the as-deposited TiN, Ta, and TaN substrates. The TiN substrate has a rougher surface than the Ta and TaN substrates, whereas the Ta substrate has the smoothest surface. Different substrate surface properties will have different effects on the nucleation processes of the Cu film, which in turn will lead to different properties of final Cu film. Figure 8 shows the SEM micrographs for Cu nucleations on TiN, Ta, and TaN substrates at 160 °C for 1 and 2 min. During the first minute,

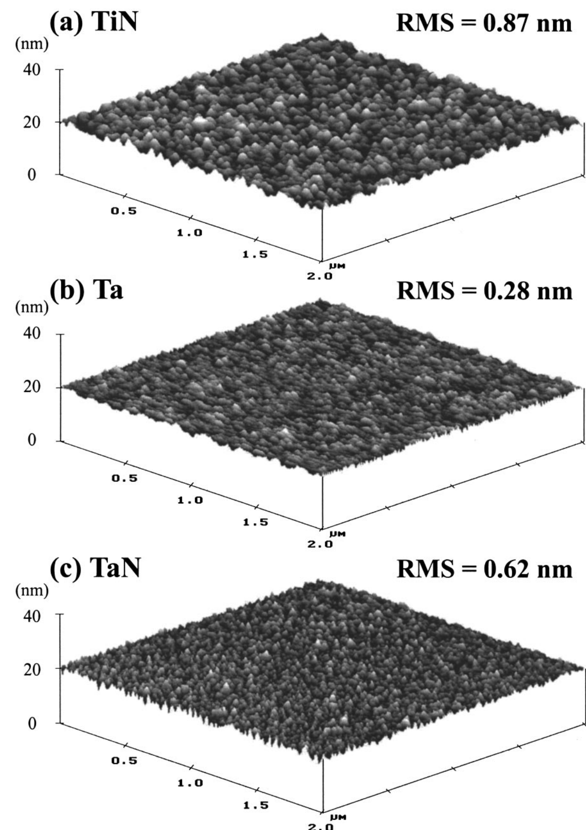

FIG. 7. AFM images showing surface morphology of (a) TiN, (b) Ta, and (c) TaN substrates.

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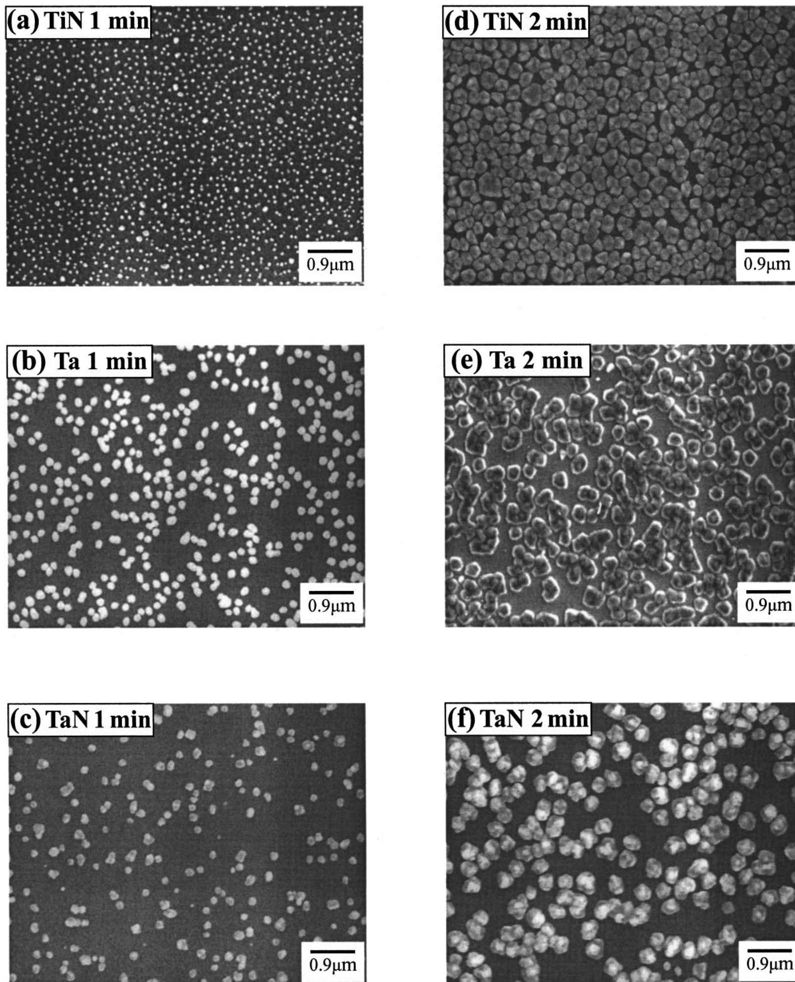

FIG. 8. SEM micrographs showing Cu nucleation at 160 °C for 1 min deposition on (a) TiN, (b) Ta, and (c) TaN substrates, and for 2 min deposition on (d) TiN, (e) Ta, and (f) TaN substrates.

the Cu-containing adspecies [Cu(hfac)] easily nucleated on the TiN substrate, forming dense, small, and uniformly distributed Cu nuclei [Fig. 8(a)]. On the other hand, the Cu-containing adspecies sparsely nucleated on Ta and TaN substrates, especially on the latter, forming less dense Cu nuclei of larger size [Figs. 8(b) and 8(c)]. After CVD for 2 min, Cu nuclei on the TiN substrate coalesced and grew into dense and large Cu grains [Fig. 8(d)]. On the Ta and TaN substrates, however, the subsequent Cu-containing adspecies tended to nucleate on the existing Cu nuclei (grains) rather than on the substrate surfaces, thus forming larger but less densely distributed Cu grains [Figs. 8(e) and 8(f)]. We presume that the Cu-containing adspecies [Cu(hfac)] are easily adsorbed by the TiN substrate and proceed with deposition of Cu via disproportionation as compared to that on Ta and TaN substrates, although the resistivity of TiN is larger than those of Ta and TaN, making it unfavorable for the process of electron exchange. However, there is a higher content of impurities adsorbed on Ta and TaN substrate surfaces at the initial stage of nucleation; this would degrade the conductivity of Ta and TaN substrate surfaces and thus adversely affect the process of electron exchange, resulting in difficult deposition of Cu films on Ta and TaN substrates. TiN film was similarly observed to be a good nucleation layer in metal-organic CVD.[23] The dense and uniformly distributed Cu grains on the TiN substrate indicate that TiN substrate has a higher surface energy or lower interfacial energy, thus resulting in a smaller wetting angle (contact angle) of Cu nuclei on TiN than that on Ta and TaN substrates.[24,25] The smaller wetting angle enhanced the Cu film growth in two dimensions (layer growth), forming the most stable and (111) closely packed configuration;[24] thus, the Cu film on the TiN substrate has a higher (111)-preferred orientation than those on Ta and TaN substrates.

Figure 9 illustrates the SEM micrographs of Cu films deposited at 160 and 200 °C for 10 min on TiN, Ta, and TaN substrates. For the films deposited at 160 °C, Cu grains appear to be agglomerate and the surface morphologies show no obvious difference for all Cu films. For the films deposited at 200 °C, however, the Cu films on TiN and Ta substrates exhibit a compact arrangement and clear boundaries of Cu grains, whereas the Cu film on the TaN substrate reveals a loose arrangement of grains mixed with different sizes. The surface roughness of Cu films deposited on various substrates was analyzed by AFM and illustrated in Fig. 10. The average surface roughnesses (rms) of the Cu films on TiN, Ta, and TaN substrates were determined to be 49.5, 34.2, and 55.9 nm, respectively. Presumably, the smoothness of the film surface is directly related to the smoothness of the substrate surface.

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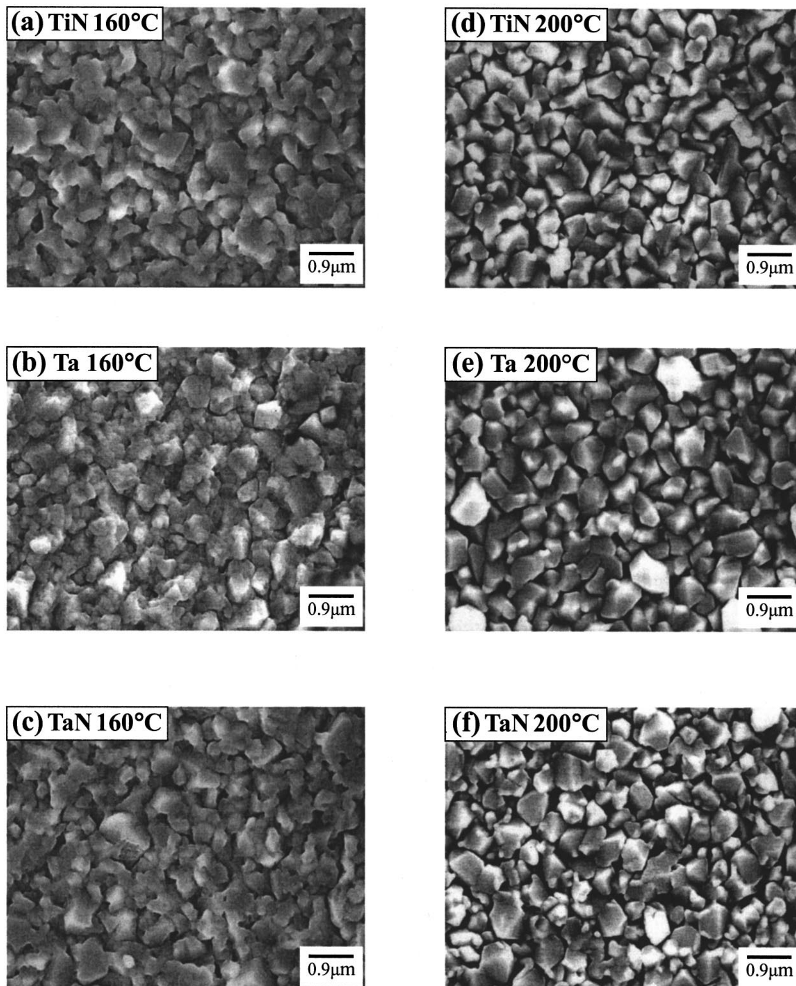

FIG. 9. SEM micrographs showing surface morphology of Cu films deposited at 160 °C on (a) TiN, (b) Ta, and (c) TaN substrates, and at 200 °C on (d) TiN, (e) Ta, and (f) TaN substrates. The deposition time is 10 min.

C. Adhesion measurement

A simple technique—the Scotch tape pulling test—was used to assess the adhesion between the CVD Cu film and the underlayer substrate. We measured the adhesion of CVD Cu films deposited on TiN, Ta, and TaN substrates at a temperature ranging from 120 to 240 °C; results of the adhesion test are summarized in Table I. All of the Cu films deposited on the TiN substrate (at 120 to 240 °C) passed the Scotch tape pulling test. For the Cu films deposited on Ta and TaN substrates, the films deposited at and above 160 °C passed the Scotch tape pulling test, whereas the films deposited at temperatures below 160 °C peeled off after the pulling test. At low deposition temperatures, there is less intermixture of Cu and the substrate materials at the interface than at high deposition temperatures.[26] Thus, Cu films deposited at low temperatures tend to have an inferior adhesion. Moreover, the surface condition of the substrate and the size, density, and distribution of Cu nuclei on the substrate surface during the initial stage of Cu CVD play important roles for film adhesion. It was reported that film adhesion can be promoted by rougher substrate surfaces because there is a larger surface area than a flat surface.[26] Because the TiN substrate has a rougher surface, as well as denser and smaller Cu nuclei on its surface, during the initial stage of nucleation than Ta and TaN substrates, the Cu film adhesive area on the TiN substrate is effectively enlarged, leading to superior adhesion of Cu films on the TiN substrate. The adhesion can be degraded by impurities (F, C, and O) absorbed on the substrate surface.[14,26] Because the impurity content at Cu/Ta and Cu/TaN interfaces are higher than that at the Cu/TiN interface, this might also contribute to the fact that a Cu film on the TiN substrate has superior adhesion.

Copper CVD possesses a superior void-free via/trench filling capability. We believe that low sticking coefficient and high re-emission frequency of Cu-containing adspecies [Cu-(hfac)TMVS] are essential to obtain good film conformality and void-free vias/trenches filling capability.[27] Because low sticking coefficient and high re-emission frequency can be achieved by decreasing the substrate temperature, Cu CVD at low temperatures is preferred to obtain a void-free filling for high-aspect-ratio vias/trenches. From this viewpoint, a TiN substrate provides a good nucleation layer for Cu CVD.

IV. CONCLUSION

Cu CVD on different substrates, including TiN, Ta, and TaN, at a pressure of 150 mTorr and a temperature ranging from 120 to 240 °C was studied with regard to the physical property, nucleation, and adhesion of the deposited Cu films.

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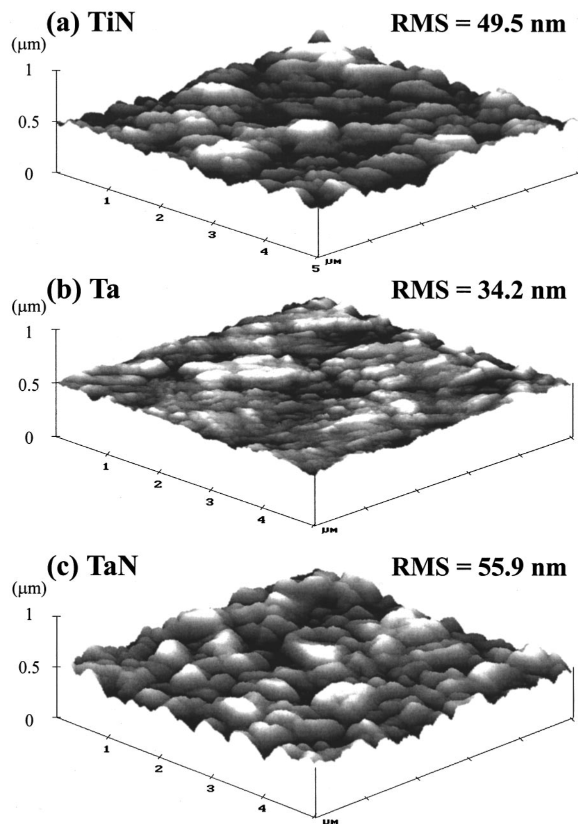

FIG. 10. AFM images showing surface morphology of Cu films deposited at 160 °C for 10 min on (a) TiN, (b) Ta, and (c) TaN substrates.

The Cu films deposited on TiN substrates have a number of favorable properties over the films deposited on Ta and TaN substrates. These include lower electrical resistivity, lower impurity contamination, and higher (111)-preferred orientation. Moreover, CVD of Cu films on TiN substrates has a shorter incubation time and better film adhesion to the underlayer substrate. Presumably, the TiN substrate has a higher surface energy than Ta and TaN substrates, and the high surface energy enhances the film growth in two dimensions (layer growth), resulting in Cu film with a higher (111)-preferred orientation. During the nucleation stage of Cu nuclei, the dense, small, and uniformly distributed Cu nuclei (grains) on the rough TiN substrate surface (which is rougher than Ta and TaN substrate) effectively enlarge the adhesive area; the enlarged adhesive area, together with the lower content and narrow distribution of impurities at the Cu/TiN interface, resulted in Cu film of superior adhesion to the TiN substrate. From the viewpoint of adhesion, microstructure, and physical property of Cu film, we may conclude that TiN is a better underlayer substrate than Ta and TaN for Cu CVD.

TABLE I. Results of the Scotch tape pulling test on the adhesion of CVD Cu films deposited at different temperatures on various substrates.

	Cu film deposition temperature (°C)						
Substrate	120	140	160	180	200	220	240
TiN	P	P	P	P	P	P	P
Ta	F	F*	P	P	P	P	P
TaN	F	F*	P	P	P	P	P

P: Cu film passed the Scotch tape pulling test.
F: Cu film peeled off after the Scotch tape pulling test.
F*: Cu film partially peeled off after the Scotch tape pulling test.

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