Articles

Surfactant-Catalyzed Chemical Vapor Deposition of Copper Thin Films

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A novel concept of chemical vapor deposition that uses a simple atomic adsorbate as a catalytic surfactant is introduced and demonstrated for deposition of copper thin films from Cu^I(hfac)(vtms). A submonolayer of iodine atoms adsorbed on the copper film surface catalyzes the surface reaction to reduce the activation energy from 15.3 kcal/mol for the uncatalyzed deposition to 6.1 kcal/mol. As a result, the growth rate is enhanced by as much as ~100 times and deposition is possible even at 50 °C with a rate of 250 Å/min. The product analysis by gas chromatography−mass spectroscopy reveals that on an I-adsorbed surface Cu^I(hfac)$_{ad}$ further dissociates into Cu and (hfac)$_{ad}$ by interacting with an I$_{ad}$ atom, and (hfac)$_{ad}$ subsequently recombines to desorb as (hfac)$_2$. For the uncatalyzed deposition, the disproportionation reaction between two $[Cu^I$(hfac)$]_{ad}$ complexes was confirmed. Iodine adatoms also act as a segregating surfactant to suppress the surface roughness, allowing deposition of 1 μm thick films with a root-mean-square roughness of 35 Å. The electrical resistivity of the films is $\rho = 2 \pm 0.2$ mΩ cm, close to the bulk value of 1.67 mΩ cm.
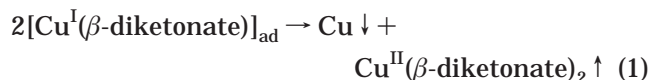
Introduction

Copper is now widely accepted as the material for interconnecting metal lines in next-generation ultra-large-scale integrated circuit devices.[1] A need for a viable deposition technology has motivated many investigations[2] on metal−organic chemical vapor deposition (MOCVD) of copper, and most of the recent studies have been performed with precursor molecules of the type Cu^I(β-diketonate)L, where L is a neutral Lewis base ligand. Although a deposition rate as high as 9000 Å/min has been achieved,[3] a typically growth rate is ≳1000 Å/min. More significantly, deposited copper films invariably have a very rough surface, which is a great disadvantage in multilevel metallization requiring further processing steps.

In thin-film growth by MOCVD, it would be ideal if a simple atom adsorbed on the substrate or on the growing film could catalyze the surface reactions involved in deposition and simultaneously could promote lateral growth to suppress the surface roughness of the film. For an adsorbate to play such a dual role of a catalytic surfactant, first it should be able to promote dissociation of the precursor molecule and desorption of the ligands by favorably interacting with the coadsorbed precursor. Second, it also has to promote 2D

lateral growth by suppressing 3D growth. Last, it must segregate out onto the growing film surface for continuous activity. On the basis of the knowledge of the surface chemistry, one may be able to find such an adsorbate appropriate to a given deposition system.

Copper MOCVD from Cu^I(β-diketonate)L precursors is known to commonly proceed via dissociative adsorption of the precursor molecule into $[Cu^I$(β-diketonate)$]_{ad}$ and L$_{ad}$ followed by a disproportionation reaction and desorption of L$_{ad}$[3,4]:

$$2[Cu^I(\beta\text{-diketonate})]_{ad} \rightarrow Cu\downarrow + Cu^{II}(\beta\text{-diketonate})_2\uparrow \quad (1)$$

$$L_{ad} \rightarrow L\uparrow \quad (2)$$

Which of these two steps is the rate-determining step (rds) has not been well established yet. It has been shown by temperature-programmed desorption that a weakly adsorbing neutral ligand L rapidly desorbs at the deposition temperature,[5] which suggests that the disproportionation reaction 1 involving migration of a bulky (β-diketonate) group may be the rds.[6] Then, one can anticipate that an electronegative atom such as a halogen atom adsorbed adjacent to $[Cu^I$(β-diketonate)$]_{ad}$

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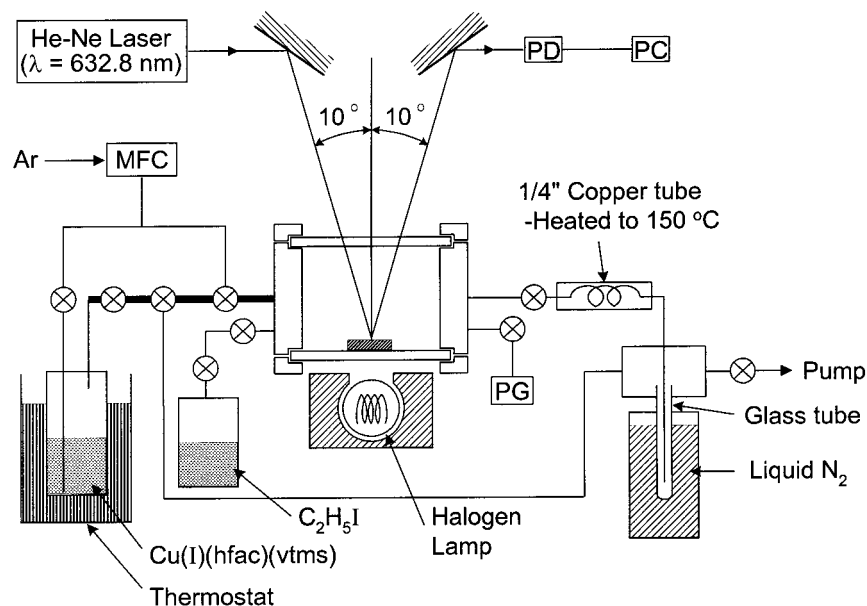
Figure 1. A schematic diagram of the copper MOCVD system: PD = photodiode, PC = personal computer, PG = pressure gauge, and MFC = mass flow controller.

may weaken the ionic $Cu^+-(\beta\text{-diketonate})^-$ bond to facilitate further dissociation of $[Cu^I(\beta\text{-diketonate})]_{ad}$ and thereby to promote the deposition rate. Among the halogen atoms, iodine is the most appropriate one because it adsorbs least strongly on copper and hence easily segregates out onto the copper film surface.[7] On an iodine-adsorbed surface, newly deposited copper adatoms would have a greater probability of binding to an iodine atom nearby to form 2D nuclei around it rather than aggregating with one another to form 3D islands. An enhanced 2D nucleus density can promote lateral growth, thus rendering the film surface smooth.[8] We demonstrate here that a submonolayer of iodine indeed acts as a segregating catalytic surfactant to allow deposition of copper films with a smooth surface at a greatly enhanced rate.

Experimental Section

Copper MOCVD was carried out with copper(I) hexafluoroacetylacetonate vinyltrimethylsilane, $Cu^I(hfac)(vtms)$, on 500 Å TiN ($\rho \approx 150$ mΩ cm) films on n-type Si(100) substrates in a cold-walled horizontal reactor schematically shown in Figure 1. A 500 Å TiN barrier layer was deposited by reactive dc magnetron sputtering of Ti in a plasma excited with N_2 and Ar at 5 mTorr. The top and bottom of the reactor were O-ring sealed with quartz plates. The 1.5×1.0 cm^2 sample was directly placed on the bottom quartz plate and radiatively heated using a halogen lamp through a metal window underneath the bottom quartz plate. The substrate temperature was carefully measured under the deposition conditions with a K-type thermocouple attached to the barrier layer using a minimal amount of a ceramic glue (Aremco 516).

The liquid precursor contained in a bubbler at 30 °C was carried into the reactor by argon carrier gas at a flow rate of 20 standard cm^3/min. The precursor delivery line was kept at 35 °C to prevent condensation. Deposition was always made under a reactor pressure of 1.5 Torr. For adsorption of iodine atoms on the film surface, ethyl iodide (C_2H_5I, bp = 72 °C) vapor in a container held at room temperature was directly

introduced into the reactor at the deposition temperatures. C_2H_5I is known to dissociatively adsorb on the copper surface resulting in prompt desorption of ethylene and hydrogen via β-hydride elimination above $\sim$230 K, while the iodine atom remains adsorbed up to 930 K.[9] The coverage of adsorbed iodine atoms was estimated to be about 0.1 monolayer (ML) (0.1 iodine atom per surface Cu atom).[10] The specular reflectivity of a He−Ne laser beam ($\lambda = 632.8$ nm) was measured at the incidence angle of 10° from the surface normal to monitor the surface roughness during the copper deposition.

The reaction products were collected by using a liquid nitrogen trap installed between the reactor and the mechanical pump. For complete conversion of the unreacted precursor molecule to the products, an oxygen-free high conductivity copper tube (0.25 in. o.d., 15 cm long) between the reactor outlet and the cold trap was maintained at 150 °C so that it also serves as an iodine-adsorbed copper substrate. The collected reaction products were analyzed by gas chromatography−mass spectroscopy (GC-MS) in the chemical ionization mode to minimize fragmentation of the parent ions. The thickness of the copper films was measured by a stylus-profilometer (α-step) after making a step by combination of photomasking and wet etching in 3 N HNO_3 solution. The sheet resistance of the film was measured with a four-point probe. The deposited copper films were characterized by X-ray diffraction, X-ray photoelectron spectroscopy (XPS), scanning electron microscopy, atomic force microscopy (AFM), and Auger depth profiling.

Results and Discussion

Figure 2 shows the Arrhenius plot for the film growth rates measured at substrate temperatures ranging from 50 to 200 °C. When deposition was carried out without iodine adsorption, the growth rate is $\sim$10 Å/min at 100 °C and increases to $\sim$800 Å/min at 200 °C. Surface-reaction-controlled deposition is evident from the straight

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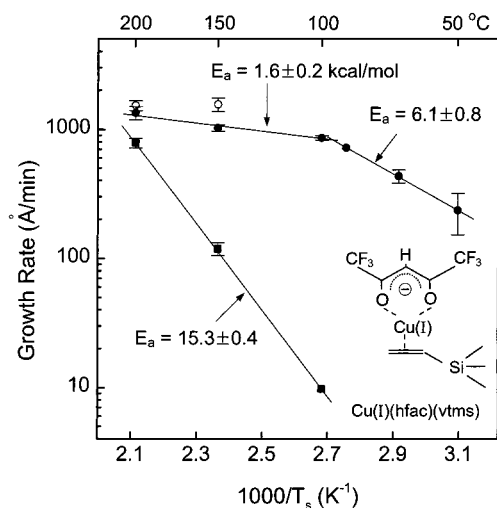(8) Sieradzki, K.; Brankovic, S. R.; Dimitrov, N. *Science* **1999**, *284*, 139.

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(10) A monolayer of methyl iodide molecularly adsorbed on Cu(111) at 100 K partially dissociates into CH_3(ad) and I(ad) upon heating to $\sim$160 K. The coverage of CH_3(ad) or equivalently of I(ad) thus formed was measured by temperature-programmed desorption and turned out to be 0.08 ML (Kim, J.-Y.; Lee, J. *J. Chem. Phys.*, in press). The maximum iodine coverage achievable by C_2H_5I exposure to Cu(111) up to saturation above 250 K was estimated as $\sim$0.1 ML on the basis of AES measurements.

Figure 2. Arrhenius plot of the deposition rates as a function of the substrate temperature (T_s). Copper films were deposited on TiN substrates (a) only with $Cu^I(hfac)(vtms)$ precursor (■), (b) with the precursor and continuously added C_2H_5I (●), and (c) only with the precursor following brief iodine adsorption in the beginning of deposition (○).
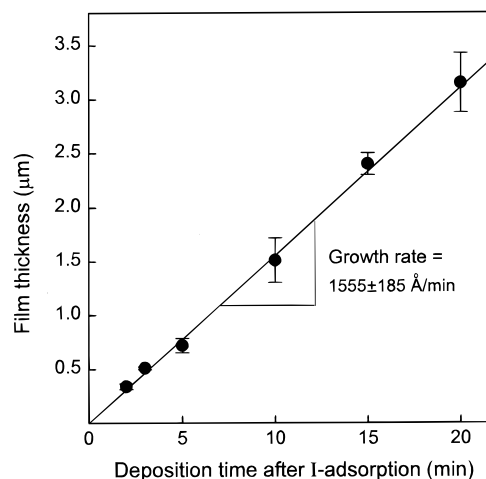

Figure 3. The thickness of copper films deposited at $T_s = 150$ °C as a function of the deposition time following brief initial iodine adsorption. The linear relationship indicates that initially adsorbed iodine atoms segregate out onto the surface of the growing copper film to exhibit a constant catalytic activity.

line with an activation energy $E_a = 15.3 \pm 0.4$ kcal/mol, which is in agreement with results reported in the literature for copper deposition from $Cu^I(hfac)(vtms)$.[11] On the other hand, simultaneous feeding of a small amount of C_2H_5I vapor (partial pressure of ~50 mTorr) greatly enhances the growth rate to 900, 1000, and 1300 Å/min at 100, 150, and 200 °C, corresponding to an enhancement factor of 90, 8.3, and 1.6, respectively. Copper films could be deposited at a temperature as low as 50 °C with an appreciable growth rate of ~250 Å/min. Note that iodine adsorption markedly lowers the activation energy to $E_a = 6.1 \pm 0.8$ kcal/mol in the surface-reaction-limited regime below 100 °C, implying that the surface reaction is somehow modified. Above 100 °C, the surface reaction is so fast that deposition is now transport-limited exhibiting only a weak temperature dependence. Thus, the enhancement factor becomes smaller with increasing temperature.

Copper deposition occurs by nucleation and growth of 3D islands followed by island coalescence to form a continuous film. We have previously shown that the onset and completion of island coalescence are distinguished by a pronounced minimum (R_{min}) and maximum (R_{max}) in the specular reflectivity, respectively.[12] To test whether continuously added C_2H_5I participates in the deposition reaction as a reactant or not, we have performed the following control experiments; first we deposited copper at 150 °C only with the precursor until spherical-cap-shaped 3D copper islands coalesce to form a continuous film, i.e., until the reflectivity reaches R_{max}. At this stage, the film thickness judged from the average island size in the SEM image of the film at the R_{min} is about 300 Å.[12] Then, the copper film was briefly exposed to C_2H_5I vapor for ~10 s and deposition was resumed after pumping out the residual C_2H_5I vapor. Hereafter, we call this mode of deposition with "brief iodine adsorption". The film thickness plotted against the

deposition time following brief iodine adsorption (Figure 3) shows a constant growth rate of 1560 Å/min. The result clearly indicates that continuous C_2H_5I addition is not necessary to obtain an enhanced growth rate, and therefore C_2H_5I is not a reactant. We thus conclude that a submonolayer of iodine atoms adsorbed on the copper surface acts as a catalyst for the deposition reaction by modifying the surface reaction of the precursor molecule. The growth rate is somewhat higher for brief iodine adsorption than for continuous addition. This could be due to the competition for the adsorption site between the precursor molecule and C_2H_5I. Other iodine-containing molecules also have exhibited essentially the same catalytic effect as long as the dissociatively adsorbed species other than the iodine atom do not remain adsorbed at the deposition temperature; for example, CH_3I resulted in the same enhancement factor as C_2H_5I at $T_s \geq 100$ °C but below 100 °C, where methyl remains adsorbed on the copper surface,[13] the enhancement factor becomes smaller due to adsorption site blocking by $CH_3(ad)$. Exposing C_2H_5I directly onto the TiN surface prior to deposition did not show any enhancement effect probably because C_2H_5I does not dissociatively adsorb on the TiN surface.

The result in Figure 3 further indicates that iodine adatoms are neither consumed by the deposition reaction nor imbedded in the growing copper film. If otherwise, prolonged deposition following brief initial iodine adsorption would result in a diminished average growth rate because of the gradual loss of catalytic activity with increasing deposition time. From this we can infer that iodine atoms segregate out onto the copper surface to maintain a continuous catalytic activity. Surface segregation of iodine was also confirmed by the X-ray photoelectron spectra (Figure 4), showing the same surface iodine coverage on the two copper films deposited for 0 and 6 min following brief iodine adsorption. A similar observation has been made previously by Stickney et al.[7] in electrochemical deposition of copper on I-preadsorbed metal surfaces. Surface segre-

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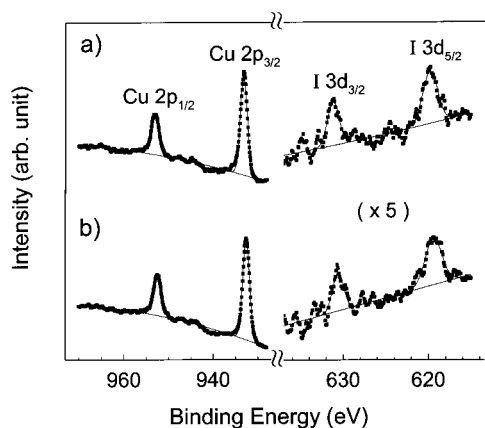(13) Lin, J.-L.; Bent, B. E. *J. Phys. Chem.* **1993**, *97*, 9713.

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Figure 4. The X-ray photoelectron spectra showing the same surface coverages of iodine atom for two copper films deposited for (a) 0 and (b) 6 min following brief iodine adsorption at the R_{max}.

Figure 5. GC-MS spectra obtained by chemical ionization method($M+CH_5^+ \rightarrow MH^+ + CH_4$) showing that $Cu^{II}(hfac)_2$ and $(hfac)_2$ are the major high mass reaction products in (a) uncatalyzed and (b) iodine-catalyzed copper deposition, respectively.

gation of iodine is believed to occur by site exchange with copper adatoms.[14]

To find out how adsorbed iodine modifies the surface reaction to lower the activation energy, we have analyzed the reaction products by gas chromatography–mass spectroscopy (GC-MS). The GC-MS spectra obtained by the chemical ionization method ($CH_5^+ + M \rightarrow MH^+ + CH_4$) for copper deposition without iodine adsorption (Figure 5a) shows the main peak at $m/e = 478$ corresponding to $[Cu^{II}(hfac)_2] \cdot H^+$ ion. This clearly confirms the disproportionation reaction (1) above. Detection of $(vtms) \cdot H^+$ at $m/e = 101$ and other related fragment peaks is also consistent with deposition via the disproportionation reaction.[3,4] In the case of deposition with brief iodine adsorption, the GC-MS spectra (Figure 5b) now shows no $[Cu^{II}(hfac)_2] \cdot H^+$ peak at all but instead shows two main high mass peaks at $m/e = 395$ and 393 which are not seen in the uncatalyzed deposition. We believe that these ions are the fragments of the parent ion $(hfac)_2 \cdot H^+$ ($m/e = 415$) which lost HF or HF + H_2 in the ionization process, respectively. The ion fragmentation is highly probable, considering that the loss of HF or HF + H_2 can further stabilize the resulting molecular ions by extended π-conjugation. Detection of the gas-phase $(hfac)_2$ product suggests that on the iodine-adsorbed copper surface $[Cu^I(hfac)]_{ad}$ further dissociates into Cu and $(hfac)_{ad}$, whereby two $(hfac)_{ad}$ recombine to desorb as $(hfac)_2$:

$$[Cu^I(hfac)]_{ad} \rightarrow Cu\downarrow + (hfac)_{ad} \qquad (3)$$

$$2(hfac)_{ad} \rightarrow (hfac)_2\uparrow \qquad (4)$$
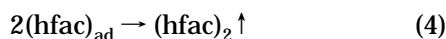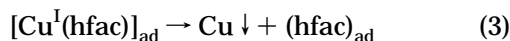
The iodine-catalyzed deposition exhibiting a relatively small activation energy of 6.1 kcal/mol can now be attributed to the modified surface reaction (3). $[Cu^I-(hfac)]_{ad}$ is a donor–acceptor complex with some ionic bonding character of $Cu^+-(hfac)^-$. Its facile dissociation on iodine-coadsorbed copper surface could be due to weakening of the ionic bond by an electronegative iodine adatom adjacent to $[Cu^I(hfac)]_{ad}$ through a direct electrostatic or indirect through-metal electronic interaction. According to the modified surface reaction mech-
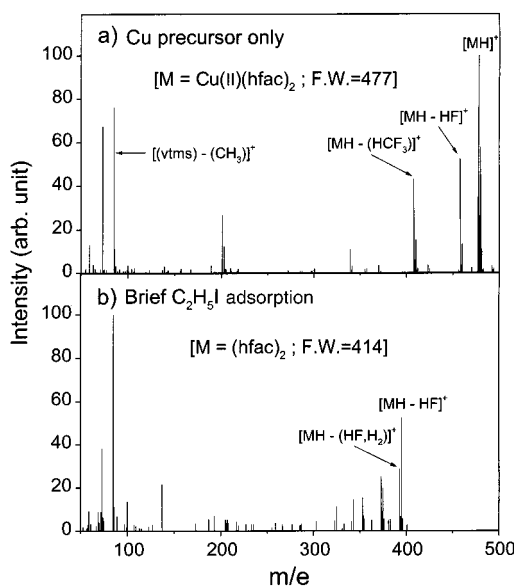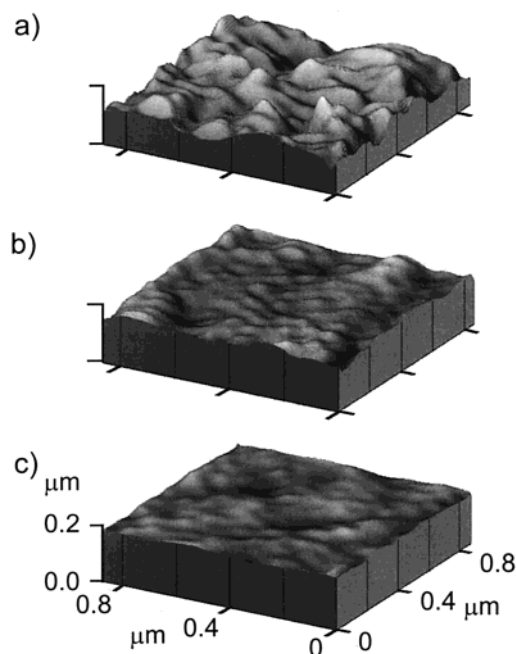

Figure 6. AFM images of $\sim 1~\mu m$ thick copper films deposited at $T_s = 150~°C$: (a) only with the precursor, (b) with the precursor and continuously added C_2H_5I, and (c) only with the precursor following brief iodine adsorption at the R_{max}. The root-mean-square surface roughness are 252, 105, and 51.5 Å, respectively.

anism, copper MOCVD with a $Cu^I(hfac)L$ type of precursor is expected to be catalyzed by adsorbed iodine regardless of the type of the ligand L.

In Figure 6 we have shown the AFM images of the surfaces of copper films deposited at 150 °C up to a thickness of 1 μm. The film grown without iodine adsorption shows a relatively rough surface with a root-mean-square (rms) roughness of 252 Å, whereas C_2H_5I addition results in a film with a rms roughness of 105 Å. A smoother surface with a rms roughness of 51.5 Å is obtained by brief iodine adsorption, which can be

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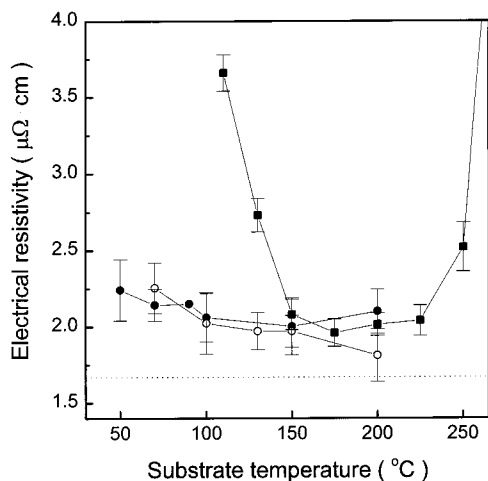
Figure 7. Electrical resistivity of the copper films deposited at various temperatures: (a) only with the copper precursor (■), (b) with the precursor and continuously added C_2H_5I (●), and (c) only with the precursor following brief iodine adsorption at the R_{max} (○).

further improved to 35 Å by lowering the temperature down to 50 °C. Considering the fact that at the stage of brief iodine adsorption the film already exhibits a surface roughness of about a few tens of angstroms, a small amount of adsorbed iodine (~0.1 ML) is remarkably effective in suppressing the surface roughness during the film deposition up to a thickness of 1 μm. The specular reflectance for the three films in Figure 6 measured with a He–Ne laser beam ($\lambda = 632.8$ nm) at the incidence angle of 10° from the surface normal is 26, 78, and 87%, respectively. This is also consistent with the AFM results. Therefore, we further conclude that iodine adatoms not only act as a floating catalyst but as a surfactant to make the copper film surface smooth. Addition of a small amount of water to Cu^I-(hfac)L (L = vtms or 2-butyne) has been reported to enhance the copper deposition rate up to five times by promoting the Cu–L bond dissociation.[15] However, oxygen was incorporated in the films at high water flow rate and no significant surface smoothing effect was observed. Thus, the role played by continuously added water is fundamentally different from that of a segregating catalytic surfactant demonstrated here.

According to the X-ray diffraction measurements,[16] the preferred orientation of the copper films deposited without adsorbed iodine depends on the substrate temperature; the dominant peak is the (200) peak at 200 °C but changes to the (111) peak. However, the copper films deposited with continuous C_2H_5I addition or with brief iodine adsorption at R_{max} are highly (111)-oriented regardless of the deposition temperature, showing a sharp (111) peak and only a small (200) peak with an intensity ratio of about 10:1. It thus appears that adsorbed iodine also promotes (111)-preferred growth of the copper film.

The electrical resistivity of the copper films estimated from the film thickness and the sheet resistance is shown in Figure 7 for films grown at various substrate temperatures with or without iodine adsorption. The films grown without adsorbed iodine show that there

exists an optimal temperature window of $150 \leq T_s \leq 200$ °C in which a low resistivity of $\rho \approx 2$ mΩ cm can be achieved. The rapid increase in ρ above 200 °C is due to interfacial void formation resulting from faceting.[12] The small increase in ρ with decreasing temperature below 150 °C has been attributed to grain boundary scattering of electrons due to the small grain size.[12] Iodine adsorption extends the temperature window to the lowest temperature tested, i.e., to $T_s = 50$ °C at which $\rho = 2.0 \pm 0.2$ mΩ cm, close to the bulk value ($\rho = 1.67$ mΩ cm). It seems that adsorbed iodine makes the grain size even larger at low temperature by enhancing the lateral growth of copper as discussed below.

It is well-established that a 3D island film growth mode can be switched to a layer-by-layer growth mode using an adsorbed layer of surfactant atoms,[17–19] in which surfactants act to enhance the nucleation density and to reduce the energy barrier associated with migration of deposited atoms down the step edge.[20–22] Very recently, Sieradzki et al.[8] have shown that atomically flat epitaxial silver layers can be electrochemically deposited on Ag(111) and Au(111) up to 150 nm in thickness using a monolayer of Pb or Cu as a mediator, in which the mediator enhances the density of 2D silver islands, thus promoting a layer-by-layer growth mode. Similarly, in copper MOCVD deposited copper atoms may diffuse and bind to iodine adatoms to form 2D copper islands around each iodine atom in a closed-packed (111) configuration. Thus, iodine adatoms acting as a seed for 2D island nucleation can enhance the density of 2D copper islands. As a result, the average size of the 2D islands is small and therefore the copper atoms deposited on top of these small 2D islands would have a greater chance to descend down the step before aggregating with one another,[23] thereby promoting 2D lateral growth. Reduction of the edge barrier for step-down migration by iodine adatoms adsorbed at step sites can further promote lateral growth. This can lead to a copper film with larger grains and a (111)-preferred texture.

Conclusions

In copper MOCVD from Cu^I(hfac)(vtms), a submonolayer of adsorbed iodine atoms on the initially formed copper surfaces catalyzes the surface reaction to enhance the growth rate as much as ~100 times. Thus, deposition is possible even at 50 °C with an appreciable growth rate of 250 Å/min. The modified surface reaction

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occurs with a low activation energy of 6.1 kcal/mol, in which further dissociation of $[Cu^I(hfac)]_{ad}$ into Cu and $(hfac)_{ad}$ is followed by recombinative desorption of $(hfac)_2$. The iodine adatoms also acts as a segregating surfactant to suppress the surface roughness by promoting lateral growth, allowing deposition of 1 μm thick film with a rms roughness of 35 Å. The new concept of using a catalytic surfactant in chemical vapor deposition demonstrated here can potentially be applied to the growth of other materials.

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