Extraction of Cu diffusivities in dielectric materials by numerical calculation and capacitance-voltage measurement

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A rigorous method of obtaining the Cu diffusivities in various SiO$_2$-based dielectric materials is proposed. The diffusion profile of Cu ions in a dielectric material is first simulated and the resulting flatband voltage shift ($\Delta V_{FB}$) is compared with the experimental results obtained by $C-V$ measurements after bias-temperature stressing (BTS). The evolution of the Cu concentration in dielectric materials is evaluated using a one-dimensional finite differential method with two unknown parameters, the diffusivity, and the maximum solid solubility of Cu ions in the dielectric material. $C-V$ measurements are conducted at 1 MHz to measure the $\Delta V_{FB}$ value of Cu/SiO$_2$/Si capacitors before and after BTS at an electric field of +1.0 MV/cm and in the temperature range between 200 and 275 °C. With this process, the Cu diffusivities in thermally grown SiO$_2$, oxynitride and SiO$_2$ deposited by plasma-enhanced chemical vapor deposition, and methyl-doped SiO$_2$ are found to be $2.22 \times 10^{-3} \exp(-1.54 \ eV/kT)$, $3.09 \times 10^{-5} \exp(-1.34 \ eV/kT)$, $2.59 \times 10^{-5} \exp(-1.18 \ eV/kT)$, and $6.07 \times 10^{-9} \exp(-0.71 \ eV/kT)$, respectively. © 2006 American Institute of Physics.

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I. INTRODUCTION

The implementation of Cu interconnects with low-$k$ dielectrics as intermetal dielectric (IMD) materials is now becoming a common practice in the field of integrated circuit (IC) processing in order to reduce the $RC$ delay time. As one utilizes Cu as an interconnecting material, one of the most serious issues which has to be dealt with is Cu migration through dielectric materials, which causes reliability problems such as the degradation of the IMD, increasing crosstalk in between the interconnects, and potentially the eventual failure of the IMD. Moreover, it has been reported that Cu migration in IMD is accelerated significantly under external electric fields, since Cu migrates in the positively charged state. Therefore, in order to guarantee the reliability of the Cu metallization, the mechanism of Cu migration in dielectric materials during bias-temperature stressing (BTS) needs to be fully understood.

Previously, we developed a general framework for the evaluation of the Cu concentration profile in dielectric materials during BTS using the one-dimensional finite differential method. This method considers that there are two driving forces for Cu migration; diffusion caused by a concentration gradient and drift caused by an electric field. The variation in the electric field across the dielectric layer due to the effect of charge screening from the migrated Cu ions was also taken into consideration by solving the Poisson’s equation. However, in order to calculate the exact diffusion profile of the Cu ions, one still has to know the diffusivity and the maximum solid solubility of the Cu ions in the dielectric materials. Unfortunately, direct determination of these parameters, for instance, by using secondary ion mass spectrometry (SIMS), turns out to be very difficult because of the dielectric charging effect, ion beam mixing into the subsurface layers, and the evolution of surface roughness during depth profiling. In this work, we propose a method of extracting the Cu diffusivity in dielectric materials by comparing the numerically calculated and experimentally measured values of the flatband voltage shift ($\Delta V_{FB}$) as a function of the BTS time. The effect of the maximum solid solubility of Cu ions in dielectric materials and the origin of the difference in Cu diffusivities in different dielectric materials are also discussed.

II. EXPERIMENT

Cu/thermally grown SiO$_2$/Si [metal oxide semiconductor (MOS)] capacitors were fabricated for $C-V$ measurement after BTS. A n-type Si (100) substrate with a resistivity of 16–23 Ω cm was chosen in order to achieve no voltage drop in Si during positive biasing at the gate metal. This condition is important for making an accurate numerical calculation of the electric potential in the SiO$_2$. A 300-nm-thick SiO$_2$ layer was thermally grown in wet ambient at 1000 °C and a Cu film with a thickness of 500 nm was deposited by thermal evaporation to prevent any plasma induced charge instabilities in the SiO$_2$ layer during metal deposition. In addition, a Ta film with a thickness of 50 nm was deposited on top of the Cu film by dc magnetron sputtering in an attempt to prevent Cu oxidation as a result of the high temperature stressing during BTS. After Ta deposition, a Ta/Cu gate was patterned in the form of a 1 × 1 mm$^2$ square dot by photolithography and subsequent wet-chemical etching. The backside electrical contact was made by thermally evaporated Al...
with a thickness of 1 μm after removing the backside oxide. Al/thermally grown SiO$_2$/Si capacitors were also fabricated as the experimental control using the same procedure used for the Cu gate capacitors, because it is known that Al does not diffuse into thermally grown SiO$_2$. The test patterns were finally annealed in forming gas ambient at 350 °C for one hour prior to BTS to minimize the effect of oxide charges other than Cu ions on the C-V characteristics and to improve the contacts of the electrodes. The room temperature C-V measurements were conducted at 1 MHz using an Agilent 4284A precision LCR meter after BTS and the subsequent cooling of the capacitors in a dark box with a Karl- sus PM-5 probe station to measure the value of $\Delta V_{FB}$ for BTS was conducted using a customized thermochuck and a Keithley 237 source measure unit in a N$_2$ purged box to prevent severe oxidation of the Cu gate and a thermochuck surface. For BTS, the temperature was varied from 200 to 275 °C and the electric field was set at +1.0 MV/cm.

III. RESULTS AND DISCUSSION

A. The capacitance-voltage characteristics of Cu/SiO$_2$/Si and Al/SiO$_2$/Si capacitors

Figure 1 shows the typical C-V characteristics for the Cu and Al gate capacitors after BTS as a function of the BTS time. The $\Delta V_{FB}$ value denoted in this figure is defined as the difference in the flatband voltage before and after BTS. In general, the $\Delta V_{FB}$ due to mobile ions in a dielectric material can be expressed as Eq. (1),

$$\Delta V_{FB} = -\frac{q}{\varepsilon_s \varepsilon_{ox}} \int_0^{t_{ox}} x \cdot C(x)dx,$$

where $q$ is the charge of an electron, $\varepsilon_s$ is the permittivity of a vacuum, $\varepsilon_{ox}$ is the relative dielectric constant of SiO$_2$, $t_{ox}$ is the thickness of SiO$_2$, $x$ is the depth of SiO$_2$ measured from the interface between metal and SiO$_2$ layer, and $C(x)$ is the Cu concentration at position, $x$.

For the Cu gate capacitors, a negative parallel shift in the C-V curve after BTS was observed, as shown in Fig. 1(a), which indicates an increase of the positive charge concentration in SiO$_2$ after BTS. The increase of inversion capacitance is observed intermittently in the C-V characteristic after BTS proceeds for 15 min at the temperature of 275 °C and the electric field of +1.0 MV/cm. This implies that Cu ions migrate near SiO$_2$/Si interface and may possibly increase the inversion capacitance by forming electrically active states at SiO$_2$/Si interface. Although further BTS could lead the abnormal C-V characteristics like low-frequency C-V characteristics might be caused by reduced effective lifetime of minority carriers in Si due to Cu ions at SiO$_2$/Si interface or in Si substrate, C-V characteristics which show parallel shift after BTS due to Cu ion migration through bulk SiO$_2$ were only considered to evaluate Cu diffusivity in bulk SiO$_2$ layer. However, for the Al gate capacitors, a saturation behavior was observed after an initial small negative shift in the flatband voltage of approximately −0.5 V, as shown in Fig. 1(b). Because it is known that Al does not diffuse into thermally grown SiO$_2$, this initial negative $\Delta V_{FB}$ is believed to originate from positive oxide charges such as alkali ions or the polarization of dielectric surfaces. The lack of spreadout of the C-V curve indicates that no interface trapped charges were generated during BTS. The effect of the charges located near the interface of metal and SiO$_2$ layer on $\Delta V_{FB}$ is quite small [Eq. (1)]. Therefore, the sole effect of Cu ions migrating into the dielectric material on $\Delta V_{FB}$ can be obtained by subtracting the value of $\Delta V_{FB}$ for the Al gate capacitors from that for the Cu gate capacitors.

Figure 2 shows the difference in the value of $\Delta V_{FB}$ between the Al and Cu gate capacitors as a function of the BTS time at various temperatures. These data clearly show that the value of $\Delta V_{FB}$ is increased as the BTS time and temperature are increased meaning that more positive charges are built up inside of SiO$_2$ with the increase of the BTS time and temperature. More close examination of the data shows that the $\Delta V_{FB}$ value initially increases quite rapidly and linearly increases with the BTS time at a given temperature, and this

![Figure 1](https://example.com/fig1.png)

**FIG. 1.** (a) C-V characteristics of Cu/thermally grown SiO$_2$/Si capacitors and (b) Al/thermally grown SiO$_2$/Si capacitors after stressing under +1.0 MV/cm at 275 °C.

![Figure 2](https://example.com/fig2.png)

**FIG. 2.** $\Delta V_{FB}$ as a function of the BTS time and temperature for thermally grown SiO$_2$ capacitors stressed at the electric field of +1.0MV/cm after subtracting the $\Delta V_{FB}$ for Al gate capacitors from that of Cu gate capacitors. Solid dots indicate measured value of $\Delta V_{FB}$ by C-V measurement after BTS and the lines indicate numerically calculated value of $\Delta V_{FB}$ with different maximum solid solubility (Cs) of Cu at each temperature.
result is consistent with other reports. As is shown in Eq. (1), the value of $\Delta V_{FB}$ is related with the charge concentration profile in the dielectric layer. The subject of this paper is then whether we can estimate the diffusivity and maximum solid solubility of Cu ions by matching the simulated values with the experimentally measured $\Delta V_{FB}$ values. This task will provide the validity of our governing equation and also provide some clues for the estimation of the diffusivity and the maximum solid solubility of Cu ions in dielectric materials and the role of each factor on the overall Cu migration through the dielectric layer.

**B. Numerical modeling of the flatband voltage shift as a function of the BTS time**

In order to figure out the effect of Cu diffusivity on the value of $\Delta V_{FB}$, we simulate several Cu diffusion profiles with different diffusivity values at constant Cs. For the calculation of the Cu concentration in SiO$_2$ and $\Delta V_{FB}$, following assumptions are made. First, the dominant diffusing species of Cu in SiO$_2$ are assumed to be singly charged positive ions (Cu$^+$) based on thermodynamic calculation of the solid solubility of various Cu species. Second, Cu migrates only normal to dielectric surface because the diffusion is significantly enhanced in parallel to the direction of electric field. It is also assumed that the Cu diffusivity in SiO$_2$ is only a function of temperature and does not depend on position and time because the maximum solid solubility of Cu in SiO$_2$ is generally too low to affect the Cu diffusivity depending on position. The detailed sequence of the numerical calculation was reported elsewhere.

Figure 3(a) shows the numerically calculated $\Delta V_{FB}$ as a function of BTS time with the different value of Cu diffusivity at the constant maximum solid solubility of Cu (Cs) in SiO$_2$. The data obtained from the simulation shows that $\Delta V_{FB}$ increases rapidly in the early stage of BTS and slowly increases with increasing BTS time and the increase of $\Delta V_{FB}$ value with the BTS time is almost linear after a certain BTS time at a given Cu diffusivity similar to experimental results. This general behavior can be attributed to the rapid increase in the Cu ion influx caused by the rapid decrease in the electric field near the Cu/SiO$_2$ interface that is induced by Cu ions migrating into SiO$_2$, as demonstrated elsewhere. It is noted that the rate of the increase in $\Delta V_{FB}$ with the BTS time $(d\Delta V_{FB}/dt)$, which is the slope of $\Delta V_{FB}$ with BTS time after initial transient time, increases as Cu diffusivity increases at the constant Cs.

Figure 3(b) shows the numerically calculated $\Delta V_{FB}$ as a function of BTS time with the different values of maximum solid solubility (Cs) at the constant value of Cu diffusivity. As Cs increases at the same diffusivity, the value of $\Delta V_{FB}$ also increases at the same BTS time maintaining the slope of $\Delta V_{FB}$ with BTS time after initial transient time. This initial rapid increase of $\Delta V_{FB}$ at the same BTS time results from the increased amount of migrated Cu ions in SiO$_2$ as Cs increases. At a certain Cs, the rate of the increase in the flatband voltage shift $(d\Delta V_{FB}/dt)$ is decreased after initial transient time because the sufficient amount of migrated Cu ions in SiO$_2$ induces the reduction of electric field near Cu/SiO$_2$ interface. It is also noted that Cs does not affect the slope of $\Delta V_{FB}$ with BTS time after initial transient time. For the sake of comparison with the experimental results, the numerically calculated value of $\Delta V_{FB}$, which was obtained as a function of the diffusivity and maximum solid solubility of Cu in SiO$_2$ as simulation parameters, is also shown in the Fig. 2.

**C. The extraction of the Cu diffusivities in dielectric materials**

As mentioned, the diffusivity and maximum solid solubility of Cu can be evaluated by comparing the value of $\Delta V_{FB}$ as a function of the BTS time obtained by experiment with that obtained by numerical calculation using Eq. (1), in which the diffusivity and maximum solid solubility, which act as the simulation parameters, were varied. From the experimental results of Fig. 2, we noted that the rate of the increase in $\Delta V_{FB}$ $(d\Delta V_{FB}/dt)$ is almost linear after a certain BTS time when we set the BTS temperature. Thus, in an attempt to minimize the fitting errors, the relationship between the rate of the increase in the flatband voltage shift $(d\Delta V_{FB}/dt)$ and the Cu ion diffusivity was used to evaluate the Cu ion diffusivity in SiO$_2$. From Eq. (1), the relationship between $d\Delta V_{FB}/dt$ and the Cu ion diffusivity in SiO$_2$ can be obtained as follows. First, $d\Delta V_{FB}/dt$, obtained from Eq. (1), can be written as

$$
\frac{d\Delta V_{FB}}{dt} = - \frac{q}{\varepsilon_0 \varepsilon_r} \int_0^{\infty} \int_0^x \frac{d}{dx} C(x,t) dx.
$$

Here, the governing equation for Cu migration in SiO$_2$ can be expressed as

$$
\frac{d\Delta V_{FB}}{dt} = - \frac{q}{\varepsilon_0 \varepsilon_r} \int_0^{\infty} \int_0^x \frac{d}{dx} C(x,t) dx.
$$
The constant value of temperature is increased.

\[ D \frac{\partial C}{\partial t} = D \left[ \cosh \left( \frac{qE}{2kT} \frac{\partial C}{\partial x} \right) - \sinh \left( \frac{qE}{2kT} \frac{\partial C}{\partial x} \right) \right], \]  

where \( D \) is the Cu ion diffusivity in SiO\(_2\), \( q \) is the charge of an electron, \( E \) is the electric field, \( b \) is the jump distance of Cu ions (\( \approx 0.27 \text{ nm} \)), \( k \) is the Boltzmann’s constant, \( T \) is the temperature (in kelvin), \( C \) is the concentration of Cu in SiO\(_2\), and \( x \) is the depth of SiO\(_2\). Because \( D \) is only a function of temperature, and not a function of the depth of the SiO\(_2\) layer \( (x) \) and time \( (t) \), and the formula inside the brackets in Eq. (3) is independent of the diffusivity at a given time, Eq. (2) can be simplified by substituting Eq. (3) \[ [dC(x,t)/dt = Df(x,t)] \] into Eq. (2), as follows:

\[ \frac{d\Delta V_{FB}}{dt} = -D \frac{q}{\varepsilon_0\varepsilon_{ox}} \int_0^{\infty} x \cdot f(x,t) dx. \]  

Therefore, it can be deduced from Eq. (4) that the Cu ion diffusivity in SiO\(_2\) is proportional to \( d\Delta V_{FB}/dt \) at a given BTS time and temperature when \( d\Delta V_{FB}/dt \) shows nearly constant value with the BTS time. The constant value of \( d\Delta V_{FB}/dt \) with the BTS time represents that the increased amount of Cu ions in SiO\(_2\) layer is constant with the BTS time. This results from the similar increase of diffusion length and amount of Cu ions in SiO\(_2\) between the same intervals of BTS time after initial transient time as illustrated in Fig. 4.

Figure 5(a) shows the value of \( d\Delta V_{FB}/dt \) as a function of temperature, which was extracted from the slope of the linear fitting line for the measured value of \( \Delta V_{FB} \) as a function of the BTS time shown in Fig. 2. In the case of 200 and 225 °C, the latter slope in Fig. 2 was used because the initial rapid increase in \( \Delta V_{FB} \) does not represent the reduction in the influx of Cu ions caused by their migration into the SiO\(_2\) layer and any uncompensated charges in the oxide. The data clearly shows that \( d\Delta V_{FB}/dt \) increases exponentially as the temperature is increased.

Figure 5(b) shows the value of \( d\Delta V_{FB}/dt \) as a function of the Cu diffusivity evaluated by a numerical calculation using Eq. (2), while varying the Cu diffusivity as a simulation parameter at different values of the maximum solid solubility \( (Cs) \). The value of \( d\Delta V_{FB}/dt \) increases linearly as Cu diffusivity increases at each Cs. Surprisingly, the calculated value of \( d\Delta V_{FB}/dt \) at different Cs values shows little change. The reason for this negligible variation in \( d\Delta V_{FB}/dt \) with Cs can be found in Fig. 4, which shows the evolution of the Cu concentration in SiO\(_2\) during BTS at different Cs values. As the Cs increases, the overall number of Cu ions migrating into the SiO\(_2\) layer increases but the diffusion length is similar. This similar diffusion length at different values of Cs suggests the negligible effect of Cs on the \( d\Delta V_{FB}/dt \) value because the \( d\Delta V_{FB}/dt \) value reflects the increased amount of Cu ions present in the SiO\(_2\) layer after a certain BTS time. Moreover, since the diffusivity is extracted from \( d\Delta V_{FB}/dt \), the effect of the Cs on the extracted diffusivity is negligible. In Fig. 5(a), the measured value of \( d\Delta V_{FB}/dt \) at 250 °C is 1.26 V/h. The Cu ion diffusivity corresponding to this value obtained from Fig. 5(b) using the numerical calculation is \( 2.48 \times 10^{-17} \text{ cm}^2/\text{s} \). When the same procedure is performed at each temperature and the corresponding diffusivity is plotted, the Cu ion diffusivity in SiO\(_2\) as a function of temperature is found to be \( 2.22 \times 10^{-3} \exp(-1.54 \text{ eV}/kT) \), as shown in Fig. 6.

Although it was expected that the temperature dependence of the maximum solid solubility would be similar to that of the diffusivity, the maximum solid solubility of Cu in thermally grown SiO\(_2\) at different temperatures showed no significant temperature dependence and had a value of \( 10^{19-20} \text{ ions/cm}^3 \) in the temperature range of 200–275 °C.
D. Cu diffusivities in different dielectric materials

For the sake of comparison, the Cu diffusivities in various dielectric materials were obtained from the literature. The Cu diffusivities in 4% phosphorous-silicate glass (4% PSG) and hydrogenated SiN (SiN: 16 at. % H) in the temperature range of 227–550 °C, which was evaluated using the 67Cu radiotracer diffusion technique, were reported to be $5.3 \times 10^{-14} \exp(-0.5 \text{ eV}/kT)$ and $4 \times 10^{-6} \exp(-1.1 \text{ eV}/kT)$ cm$^2$/s, respectively. The Cu diffusivity in thermally grown SiO$_2$ evaluated by theoretical calculation and drift time measurements were $2.5 \times 10^{-2} \exp(-1.2 \text{ eV}/kT)$ and $2.5 \times 10^{-8} \exp(-0.93 \text{ eV}/kT)$, respectively. We also extracted the Cu diffusivities in several dielectric materials using the proposed method. For Cu migration through plasma-enhanced chemical vapor deposition (PECVD) dielectrics and methyl-doped SiO$_2$, the measured $\Delta V_{FB}$ as a function of the BTS time was obtained from reports by Loke et al. and Cui et al., respectively. The Cu diffusivities in PECVD oxynitride (SiON), PECVD SiO$_2$, and methyl-doped SiO$_2$, which were determined using the same procedure used for thermally grown SiO$_2$, were $3.09 \times 10^{-5} \exp(-1.34 \text{ eV}/kT)$, $2.59 \times 10^{-5} \exp(-1.18 \text{ eV}/kT)$, and $6.07 \times 10^{-9} \exp(-0.71 \text{ eV}/kT)$, respectively. These results are summarized in Fig. 6.

Figure 7 clearly shows that in each case, the Cu diffusivities are strongly dependent on the quality of the host dielectric material. In particular, the chemistry, structure, composition, density of the dielectric material, and thermal history during the growth of the dielectric films appears to affect the Cu diffusivity in the dielectric material. Moreover, these properties are closely related to the growth techniques employed. While the diffusion mechanism in amorphous materials is more difficult to characterize than that in crystalline materials because of the lack of a regular long-range order, the Arrhenius behavior of the diffusivity of various dielectric thin films, as shown in Fig. 7, indicates that only one type of diffusion mechanism is likely to be rate controlling. Therefore, in this scheme, assuming that the mechanism of Cu diffusion in amorphous materials is analogous to that in ionic crystalline materials, the activation energy of Cu diffusion in different dielectric materials was analyzed according to the Anderson and Stuart model which was based on the classical ideas of ionic crystal theory and elasticity theory. In this model, the activation energy for ion diffusion in SiO$_2$ was assumed to be the sum of the network strain energy generated by the enlargement of the network due to the diffusing ions and the electrostatic energy between the diffusing ions and the SiO$_2$ lattice. According to this model, the activation energy has a larger value for dielectric materials with a large shear modulus, a dense network structure, and a low dielectric constant. In general, the shear modulus, density, and dielectric constant of the dielectric materials shown in Fig. 7 decrease in the following order: SiN, SiON, SiO$_2$, and SiOCH. The Cu diffusivities evaluated using the present method, which are indicated by open dots in Fig. 7, are in good agreement with this sequence. The discrepancy between the Cu diffusivities in different thermally grown SiO$_2$ and CVD SiO$_2$-based materials might be the result of the variation in the structural relaxation of SiO$_2$ depending on the growth temperature and the H content in the CVD film.

In the case of porous dielectrics, other mechanism due to structural porosity can also affect the overall migration behavior of Cu in dielectrics. If the surface diffusion inside pores or Cu clustering in porous dielectrics or structural and chemical changes of porous dielectrics occurs, Cu diffusivity can show non-Arrhenius type behavior. Therefore, the other possible diffusion path such as surface of pores and the interaction of diffusing species, which have the various charged states such as neutral Cu and Cu$^{2+}$, with dielectric matrix need to be considered to evaluate the Cu diffusivity more rigorously. However, the Arrhenius type behavior of Cu diffusivity with the single activation energy was observed even in the porous low-$k$ dielectric which was used to
evaluate Cu diffusivity in methyl-doped SiO\textsubscript{2} in this work. It is believed that the small porosity and interconnectivity of pores will contribute this behavior because the interaction of diffusing Cu ions with oxide lattice is relatively small compared with those in highly porous low-\textit{k} dielectrics. When Cu migrates significantly into dielectrics such as highly porous low-\textit{k} dielectrics, the maximum solid solubility of Cu in dielectric materials may change because dielectrics undergo structural change during Cu migration. In this case, additional physical model will be required to explain the actual situation such as clustering of Cu inside of pores and degradation and failure mechanism of porous dielectric itself during BTS rather than simple transport model.

IV. CONCLUSION

The validity of the proposed method for extracting the Cu diffusivities in thermally grown SiO\textsubscript{2} was demonstrated by comparing the numerically calculated values of $\Delta V_{FB}$ as a function of the BTS time with the measured values obtained by \textit{C-V} measurements after BTS. Although the maximum solid solubility of Cu in thermally grown SiO\textsubscript{2} evaluated in this study showed no significant temperature dependency, the evaluation of the Cu diffusivities in different dielectric materials was successful based on the proportionality of $d\Delta V_{FB}/dt$ with the diffusivity. Considering that the effect of the drift induced by the concentration gradient on the overall Cu migration is not negligible compared with that of the drift induced by the electric field under low stress conditions, it is believed that the Cu diffusivities extracted using the present method in different dielectric materials, which take into consideration both driving forces, can be used as a quantitative parameter to describe the actual situation under normal device operating conditions.

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\textsuperscript{1}International Technology Roadmap for Semiconductors, Interconnects, 45 (2005); \url{http://www.itrs.net/Links/2005ITRS/Interconnect2005.pdf}