

Interaction between copper and point defects in silicon irradiated with 2-MeV electrons

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(Received 27 March 1995)

The interaction between copper and irradiation-induced defects in silicon has been investigated with the use of Schottky-barrier structures formed by the deposition of copper on *n*- and *p*-type silicon irradiated with 2-MeV electrons at room temperature. It is found that the annealing rate of the divacancy (V_2) centers is anomalously high at temperatures below 150°C in both *n*- and *p*-type silicon. This is attributed to passivation of the V_2 centers by the highly mobile Cu through the formation of neutral complexes. A prominent level located 0.52 eV above the valence-band edge (E_v) appears in *p*-type silicon after Cu deposition at room temperature. It is suggested that this level originates from a complex formed by reaction between Cu and the carbon-oxygen centers which give rise to the "ordinary" irradiation-induced level at $E_v + 0.35$ eV. This latter level, which is normally stable up to temperatures of ~350°C, anneals rapidly at 100°C through the formation of the Cu-associated level at $E_v + 0.52$ eV which disappears upon silicide formation. Furthermore, the present results show that no injection of Si self-interstitials into the silicon substrate takes place during the formation of the metal-rich silicide η' -Cu₃Si, contrary to the conclusions of recent studies.

I. INTRODUCTION

Among all 3*d* transition metals, copper has an unusually high diffusion coefficient in crystalline silicon.¹ It diffuses as a positively charged interstitial atom, as confirmed by its drift in an electric field² and by ground-state total-energy calculations,³ with a diffusion coefficient of $\sim 10^{-8}$ cm²s⁻¹ at room temperature. In *p*-type silicon, it has been theoretically established that copper, like hydrogen, passivates shallow-acceptor impurities (e.g., B and In).⁴ However, in contrast to hydrogen, which bridges a B-Si bond and forms a stronger H-Si bond, leaving a neutralized, threefold-coordinated acceptor,^{5,6} potential-energy surface calculations⁴ place the copper atom at the antibonding site to the boron atom and passivation results from the formation of a Cu-B covalent bond (here Cu is moved away from the tetrahedral interstitial site towards the substitutional boron). These theoretical results are supported by experiments^{7,8} showing that Cu forms pairs with B and In atoms and thereby passivates their electrical activity. It has also been shown⁷ that this pair formation reduces the diffusion coefficient of interstitial copper in *p*-type silicon and results in an activation energy of 0.15 eV, which is supported by the measured dissociation energy (0.7–0.9 eV) of Coulombic-bound Cu-acceptor pairs.

In analogy with hydrogen, copper is anticipated to passivate not only shallow-acceptor impurities in silicon, but also irradiation-induced vacancy-type defects involving broken bonds.^{9,10} So far, however, very little effort has been directed at the understanding of the interaction between Cu and irradiation-induced defects in silicon, despite its potential applications in Si and GeSi/Si heterojunction devices.¹¹

In this work, Schottky-barrier (SB) structures formed by the deposition of Cu on *n*- and *p*-type silicon irradiated with 2-MeV electrons at room temperature are used to study the interaction between Cu and the irradiation-induced defects. We find that the divacancy (V_2)-related deep-levels anneal rapidly at temperatures below 150°C in both *n*- and *p*-type samples and attribute this anomalous annealing behavior to passivation of the V_2 centers by the highly mobile Cu through the formation of neutral complexes. We also show that, in addition to the two "ordinary" irradiation-induced levels in *p*-type silicon at 0.23 and 0.35 eV above the valence-band edge (E_v), a third major level located at $E_v + 0.52$ eV appears after Cu deposition at room temperature, and suggest that this level originates from a complex formed by a reaction between Cu and the carbon-oxygen centers giving rise to the $E_v + 0.35$ eV level. This latter level, which is normally stable up to ~350°C in Czochralski (Cz) silicon, anneals rapidly at 100°C through the formation of the Cu-associated level at $E_v + 0.52$ eV, which disappears upon silicide formation. Furthermore, our investigation gives additional insight into the role of metal-rich silicide formation in the injection of point defects (e.g., Si self-interstitials) into silicon.

II. EXPERIMENTAL PROCEDURE

Samples were cut from Cz silicon wafers grown in the $\langle 100 \rangle$ direction and doped with phosphorus and boron to a concentration of $\sim 1 \times 10^{15}$ to 1×10^{16} cm⁻³. The concentrations of interstitial oxygen and substitutional carbon were measured by infrared-absorption spectroscopy and found to be 5.5×10^{17} and below 1×10^{16} atoms/cm³, respectively. The samples were irradiated by

2.0-MeV electrons at nominal room temperature ($\leq 30^\circ\text{C}$) to doses in the range of $1 \times 10^{15} - 1 \times 10^{17} \text{ cm}^{-2}$. A low current density ($\leq 1 \mu\text{A}/\text{cm}^2$) was used in order to avoid beam heating effects. The sample surfaces were then cleaned using a standard chemical cleaning procedure, which included a final dip in diluted hydrofluoric acid¹² immediately before SB structures were prepared by deposition of $\sim 1000\text{-\AA}$ -thick Cu films on the samples. The Cu films were deposited using electron-beam evaporation in a pressure of $\sim 1 \times 10^{-7}$ Torr at a rate of $10 \text{ \AA}/\text{s}$, with the samples kept at room temperature. The deposition was made through a metal mask defining two rows of diodes of 0.75 and 1 mm in diameter. Annealing of the samples was performed in an inert atmosphere for 30 min at temperatures between 100 and 200°C .

For sample analysis, deep-level transient spectroscopy (DLTS) and capacitance-voltage ($C-V$) measurements were undertaken at temperatures between 80 and 290 K. In the DLTS measurements eight traditional spectra with rate windows in the range of $(20 \text{ ms})^{-1}$ to $(2560 \text{ ms})^{-1}$ were recorded during a single temperature scan. The measurements were performed under reverse-bias conditions, and no forward injection was applied. The experimental setup has been described in detail elsewhere.¹³ Trap concentration versus depth profiles were determined using a single rate window, and the temperature was held at the maximum of the studied peak within $\pm 0.5 \text{ K}$. The steady-state reverse-bias voltage was kept constant, while gradually increasing the amplitude of the majority-carrier pulse, and the depth profiles were extracted from the dependence of the DLTS signal on the pulse amplitude.¹⁴ X-ray photoelectron spectroscopy (XPS) and secondary-ion mass spectrometry (SIMS) were used to monitor the interfacial reaction between Cu and Si upon annealing, and the silicide phase formed was identified by electron diffraction.

III. RESULTS

DLTS spectra from two p -type samples irradiated to a dose of $1 \times 10^{15} \text{ cm}^{-2}$ and formed with Cu and Ti SB contacts are compared in Fig. 1(a). In the Ti (control) samples only the two "ordinary" irradiation-induced levels at $E_v + 0.23 \text{ eV}$ and $E_v + 0.35 \text{ eV}$ are observed with capture cross sections of $\sim 10^{-14}$ and $\sim 10^{-15} \text{ cm}^2$, respectively. The former level is stable at temperatures up to $\sim 200^\circ\text{C}$ and is attributed to the singly positive charge state of V_2 .¹⁵⁻¹⁷ The latter peak has been extensively studied by several authors¹⁶⁻²⁰ and found to arise from two centers, both of which are basically carbon-oxygen related defects, but with different atomic configurations (one of them the interstitial carbon-interstitial oxygen complex, C_iO_i). In the Cu samples, a third major level appears at $E_v + 0.52 \text{ eV}$ with a capture cross section of $\sim 1 \times 10^{-13} \text{ cm}^2$ (which is typical of a Coulomb attractive center), according to extrapolation of the Arrhenius plot in Fig. 2. In addition to these three levels, a relatively weak peak located at $\sim E_v + 0.24 \text{ eV}$, with a capture cross of $\sim 1 \times 10^{-15} \text{ cm}^2$, is also observed. This level disappears upon annealing at temperatures around 200°C . In Fig. 1(b), we also show DLTS spectra of a Cu sample and a

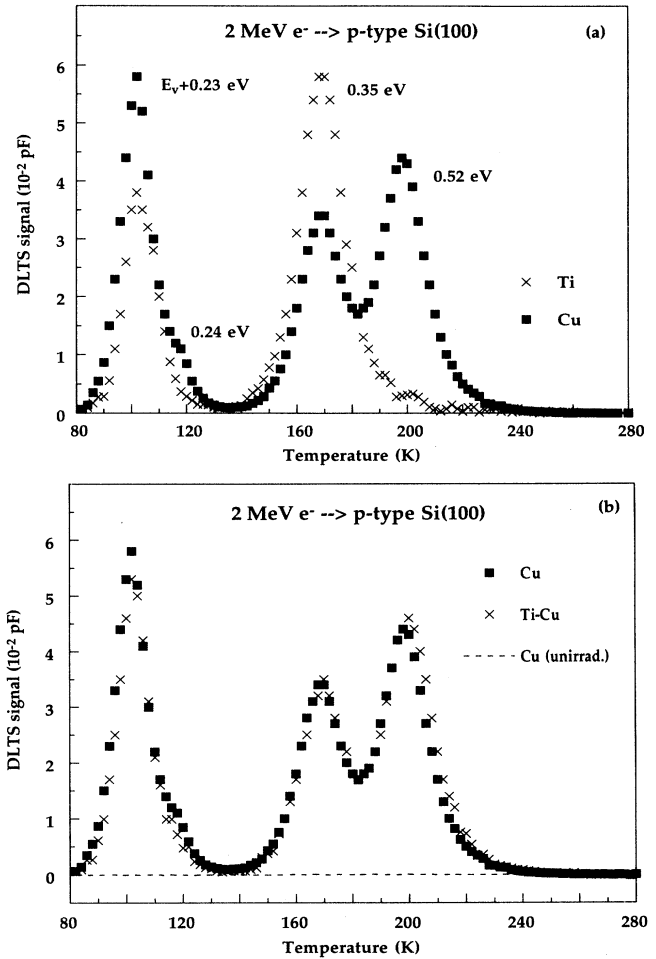


FIG. 1. (a) DLTS spectra of two p -type Si samples irradiated with 2-MeV electrons to a dose of $1 \times 10^{15} \text{ cm}^{-2}$ and formed with Cu and Ti SB contacts. (b) DLTS spectra of a p -type Si sample formed with Cu SB contacts, a p -type sample where Cu was first deposited and then chemically removed prior to the deposition of Ti SB contacts (denoted as Ti-Cu), and an unirradiated p -type Si sample formed with Cu SB contacts. [Rate window $= (1.28 \text{ s})^{-1}$.]

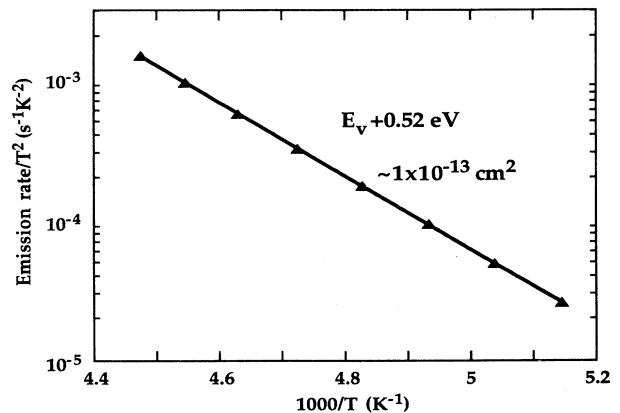


FIG. 2. Arrhenius plot of the Cu-associated level appearing after Cu deposition at room temperature on electron-irradiated p -type samples.

sample where Cu was first deposited and then chemically removed before the deposition of Ti contacts. The two spectra are identical and the level at $E_v + 0.52$ eV is now observed in the Ti-Cu samples. This indicates strongly that the defect giving rise to the $E_v + 0.52$ eV level is a Cu-associated defect. In unirradiated n - and p -type samples with Cu contacts, no levels with concentrations above 10^{11} cm $^{-3}$ are observed [Fig. 1(b)].

Depth concentration profiles of the levels at $E_v + 0.35$ and $E_v + 0.52$ eV in the as-deposited Cu samples are depicted in Fig. 3(a). The two profiles are identical in shape and resemble that anticipated for point defects induced by high-energy electrons.^{21,22} At depths greater than ~ 2.8 μ m, the concentration reaches a constant value, while a gradual decrease occurs towards the surface, and at 2 μ m the concentration is $\sim 80\%$ of that in the Si bulk.

Figure 4 shows DLTS spectra of Cu samples in the as-deposited state and after annealing at 100 and 200 $^{\circ}$ C. [$E_v + 0.23$ eV], [$E_v + 0.35$ eV], and [$E_v + 0.52$ eV] (brackets denote concentration values) decrease drastically at 100 $^{\circ}$ C. However, a substantial concentration of the $E_v + 0.23$ and the $E_v + 0.35$ eV levels still persists after the 200 $^{\circ}$ C anneal, where the Cu film has fully reacted with Si to form a metal-rich silicide phase (which has

been identified to be the η' -Cu $_3$ Si phase with a tetragonal crystal structure) as revealed by SIMS and XPS measurements.^{8,23} The $E_v + 0.52$ eV level disappears at ~ 200 $^{\circ}$ C, and two relatively weak overlapping levels at $\sim E_v + 0.49$ and $\sim E_v + 0.57$ eV emerge. The two latter levels have capture cross sections of $\sim 10^{-15}$ and $\sim 10^{-14}$ cm 2 , respectively, and originate from complexes involving Cu. In addition, a level located at $E_v + 0.29$ eV appears at about 150 $^{\circ}$ C and disappears upon further annealing at about 400 $^{\circ}$ C. Kimerling¹⁶ and Mooney *et al.*¹⁷ also reported a similar observation in electron-irradiated p -type samples and interpreted the $E_v + 0.29$ eV level to be associated with a vacancy-oxygen-boron complex. Depth profiles of [$E_v + 0.35$ eV] and [$E_v + 0.52$ eV] observed in the Cu samples after an anneal at 150 $^{\circ}$ C are presented in Fig. 3(b). The profiles change dramatically and show a considerable reduction in the concentrations of the two levels in the surface region.

In Fig. 5, we compare the isochronal annealing of the $E_v + 0.23$ eV level in p -type samples and the level located 0.43 eV below the conduction-band edge (E_c) in n -type samples formed with Cu contacts with that in p - and n -type control samples formed with Ti and Pt contacts, respectively. In moderately doped n -type Cz silicon (resistivity ≥ 1 Ω cm) the $E_c - 0.43$ eV level originates predominantly from the singly negative charge state of V_2 , while in more highly doped Cz material the overlapping level caused by the vacancy-phosphorus center (the E center) becomes of major importance.^{16,22,24} The contribution of V_2 to the level at $E_c - 0.43$ eV, with a capture cross section of $\sim 2 \times 10^{-15}$ cm 2 , was determined by a comparison with the level at $E_c - 0.23$ eV, originating from the doubly negative charge state of V_2 ; these two levels exhibit a close one-to-one proportionality in moderately doped Cz samples irradiated with MeV electrons.^{22,25} The relative contribution of V_2 to [$E_c - 0.43$ eV] is found to be more than 80% in our samples. Thus, the level at $E_c - 0.43$ eV is clearly dominated by V_2 . [$E_v + 0.23$ eV] and [$E_c - 0.43$ eV] decrease rapidly at

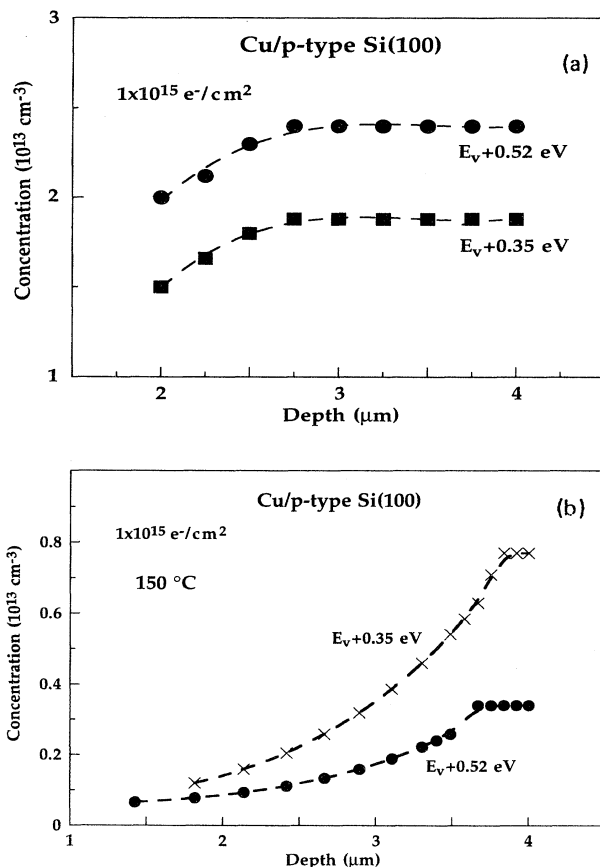


FIG. 3. Depth concentration profiles of the levels at $E_v + 0.35$ and $E_v + 0.52$ eV in electron-irradiated p -type Si samples obtained with Cu SB contacts (a) in the as-deposited state and (b) after a 30-min anneal at 150 $^{\circ}$ C.

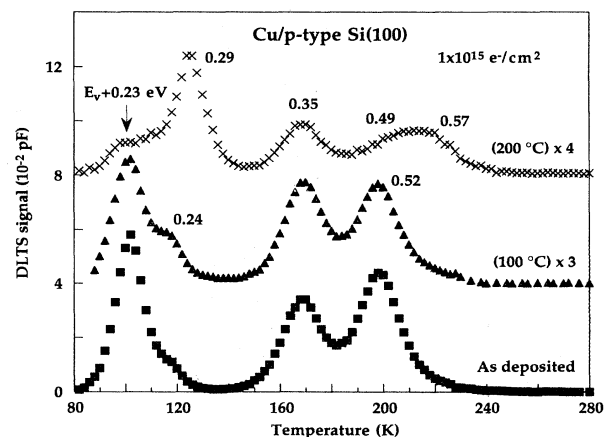


FIG. 4. DLTS spectra of electron-irradiated p -type Si samples obtained with Cu SB contacts in the as-deposited state and after a 30-min anneal at 100 and 200 $^{\circ}$ C. The two latter spectra are displaced for clarity. [Rate window = $(1.28$ s) $^{-1}$].

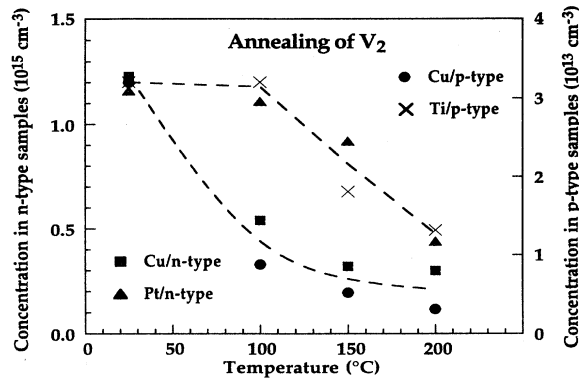


FIG. 5. Isochronal annealing (30 min) of the divacancy centers in *n*- and *p*-type Si samples irradiated with 2-MeV electrons to a dose of 1×10^{17} and $1 \times 10^{15} \text{ cm}^{-2}$, respectively, before deposition of Cu, Pt, and Ti contacts to form SB structures.

temperatures below 150°C in the Cu samples, but the annealing rate of V_2 decreases at higher temperatures; the anneal is not a first-order (exponential) process, and at 200°C , where η' - Cu_3Si is formed, [$E_v + 0.23 \text{ eV}$] and [$E_c - 0.43 \text{ eV}$] are $\sim 80\%$ and only $\sim 30\%$ lower than in the Ti and Pt samples, respectively.

However, for the control (e.g., Pt) samples a first-order process may be assumed, and an activation energy of $\sim 1.2 \text{ eV}$ and a frequency factor of $\sim 3 \times 10^9 \text{ s}^{-1}$ are obtained. These values are in reasonable agreement with those previously reported for the annealing behavior of V_2 in Cz silicon^{15,16,26} and indicate that the dominant annealing process is a long-range diffusion of V_2 and subsequent annihilation through reactions with impurities and other defects in the silicon lattice.²⁶ In this context, it may be emphasized that the annealing rate of V_2 in Cz silicon is substantially higher than in high-purity float-zone samples, where V_2 is stable up to temperatures of around 300°C .^{15,16} This difference is most likely due to the fact that the concentration of annihilation centers (impurities and defects) is several orders of magnitude higher in the Cz silicon.

Figure 6 compares the isochronal annealing of the level at $E_v + 0.35 \text{ eV}$ in *p*-type samples formed with Cu and Ti contacts. A rapid decrease of [$E_v + 0.35 \text{ eV}$] is observed at 100°C in the Cu samples, and at 200°C the concentration is $\sim 80\%$ lower than in the Ti (control) samples. The $E_v + 0.35 \text{ eV}$ level is normally stable up to $\sim 350^\circ\text{C}$ and the data for the Ti samples agree essentially with that previously reported in the literature.^{16,17}

IV. DISCUSSION

The data in Fig. 5 clearly demonstrate that in both *n*- and *p*-type silicon contacted with Cu, the annealing rate of the V_2 centers is anomalously high at temperatures below 150°C . Theoretical and experimental studies^{4,7,8} have shown that copper, like hydrogen, passivates shallow acceptors in silicon, and in analogy with hydrogen

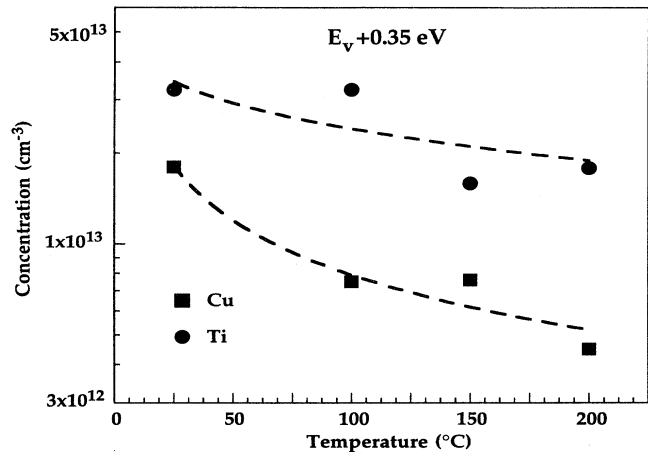


FIG. 6. Isochronal annealing (30 min) of the level at $E_v + 0.35 \text{ eV}$ in *p*-type Si samples, irradiated with 2-MeV electrons to a dose of $1 \times 10^{15} \text{ cm}^{-2}$ before deposition of Cu and Ti contacts to form SB structures. The concentration values are taken at a depth of $5 \mu\text{m}$.

passivation of vacancy-type defects with dangling bonds such as vacancy-oxygen (VO) and V_2 centers in both *n*- and *p*-type silicon,^{9,27} we suggest that copper also passivates the V_2 centers through the formation of neutral complexes. This is consistent with the annealing kinetics showing a rapid decrease in the concentration of V_2 at temperatures below 150°C , followed by a reduction in the annealing rate at higher temperatures and not a first-order process as has been reported previously.^{15,16,26} Above 150°C silicide formation, or the formation of the energetically favorable $\text{Cu}(d)\text{-Si}(p)$ bonding²⁹ causes the Cu to become less effective in the passivation process. The annealing kinetics may then be interpreted as a sum of two competing processes; one "ordinary" (first-order) annealing reaction and one "extraordinary," where V_2 centers are regenerated. A candidate for the latter process is dissociation of the Cu-passivated V_2 complexes ($\text{Cu}V_2 \rightarrow \text{Cu} + V_2$), and as a result, electrically active V_2 centers reappear giving rise to an almost constant net concentration of V_2 above 150°C . Here, it should be pointed out that there is evidence also for Cu passivation of the electrical activity of the VO centers and formation of neutral complexes.²⁸

The level at $E_v + 0.52 \text{ eV}$ appears in irradiated *p*-type silicon after Cu deposition at room temperature. The energy position is identical to that previously reported in the literature,^{29,30} for a Cu-associated level in *p*-type material, where Cu was introduced by diffusion at a high temperature followed by rapid quenching, as measured by Hall effect, photoconductivity, and DLTS measurements. However, the present level anneals out already at $\sim 200^\circ\text{C}$ and is, therefore, not believed to originate from the same center as the level observed in the Cu-diffused silicon. Normally, DLTS spectra of electron-irradiated *p*-type Cz silicon are dominated by the level at $E_v + 0.35 \text{ eV}$,¹⁶⁻¹⁸ while in the present Cu samples, the V_2 -related level at $E_v + 0.23 \text{ eV}$ is the major peak. If the two spec-

tra in Fig. 1(a) are normalized, with respect to the amplitude of the V_2 peak, it is interesting to note that $[E_v + 0.35 \text{ eV}]$ in the Ti samples and the sum of $[E_v + 0.35 \text{ eV}]$ and $[E_v + 0.52 \text{ eV}]$ in the Cu samples are identical within 10%. Thus, a possible identification of the $E_v + 0.52 \text{ eV}$ level is a complex resulting from interaction between Cu and the carbon-oxygen centers giving rise to the $E_v + 0.35 \text{ eV}$ level, and its growth is accompanied by a corresponding reduction in $[E_v + 0.35 \text{ eV}]$. This identification is also consistent with the identical shape of the depth profiles for $[E_v + 0.35 \text{ eV}]$ and $[E_v + 0.52 \text{ eV}]$ suggesting that Cu diffuses at room temperature and reacts with the stable carbon-oxygen complexes (Fig. 3).

The $E_v + 0.35 \text{ eV}$ level is normally stable up to temperatures of $\sim 350^\circ\text{C}$, where the carbon-oxygen centers start to dissociate. Isothermal annealing studies of the infrared-absorption bands associated with the centers reveal a decay with first-order kinetics and an activation energy of $\sim 2.0 \text{ eV}$.³¹ The preexponential factor is $\sim 3 \times 10^{13} \text{ s}^{-1}$, i.e., of the order of the frequency of one atomic vibration, strongly suggesting that the destruction occurs by a 'simple' breakup or dissociation. However, in the Cu samples a rapid anneal is observed already at 100°C . This is attributed to the formation of the Cu-associated level at $E_v + 0.52 \text{ eV}$, which disappears upon annealing at $\sim 200^\circ\text{C}$, indicating that silicide formation, or the formation of the Cu(*d*)-Si(*p*) bonding destroys the Cu-associated complex. In contrast to the results for V_2 , there is no evidence for regeneration of the $E_v + 0.35 \text{ eV}$ level after silicide formation (Fig. 6). This implies that the original configuration of the carbon-oxygen centers giving rise to the $E_v + 0.35 \text{ eV}$ level is not restored and Cu has promoted a low-temperature annealing of these centers. Some controversy has existed about their atomic configuration, but it seems now firmly established that at least 80% of the defects causing the $E_v + 0.35 \text{ eV}$ level are interstitial carbon-interstitial oxygen (C_iO_i) complexes with an overcoordinated oxygen atom.^{17,19,32} Destruction of the C_iO_i centers at low temperatures can be achieved by continuing the electron irradiation; at high enough electron doses the concentration of C_iO_i centers decreases, presumably by capturing of Si self-interstitials and formation of optically and electrically inactive $C_iO_i + I_n$ complexes.³³ However, as discussed in the following paragraph, we find no evidence for the generation of Si self-interstitials during silicide formation. One possibility may be that stable formation of Cu(*d*)-C(*p*) bonding occurs at $\sim 150^\circ\text{C}$, and the electrical activity corresponding to C_iO_i , which arises from the unpaired electron predominantly residing on the carbon atom in a *p* orbital, ceases to exist. On the other hand, Cu-C bonding is anticipated to be weak, and further work is needed to elucidate the exact role of Cu in the low-temperature annealing of the C_iO_i centers.

Let us now consider the role of silicide formation in the injection of Si self-interstitials into silicon in view of the anomalous annealing behavior of the V_2 centers. Recently, Ronay and Schad³⁴ reported that the formation of metal-rich silicides, where the metal is the dominant

moving species during silicidation, generates self-interstitials in silicon. This suggestion is examined here by monitoring the concentration of V_2 centers in both *n*- and *p*-type silicon during formation of η' -Cu₃Si¹⁰. The V_2 centers are normally stable up to $\sim 200^\circ\text{C}$ in Cz silicon, and a well-established annihilation reaction of the V_2 centers is the recombination with Si self-interstitials ($V_2 + I \rightarrow V$).³⁵ In fact, these annihilation processes have a high efficiency, e.g., only 10% of the vacancies and interstitials generated by high-energy electrons are estimated to escape direct recombination.³⁶ However, in the temperature range $150\text{--}200^\circ\text{C}$, where the metal-rich silicide η' -Cu₃Si is formed, the concentration of the $E_c - 0.43$ and $E_v + 0.23 \text{ eV}$ levels remains almost constant, and no abrupt annihilation of V_2 takes place (Fig. 5). It should also be pointed out that both the Pt and Ti contacts are expected to be stable in the temperature range studied since the formation rate of Pt- and Ti-rich silicides is very small (less than $10^{-17} \text{ cm}^2/\text{s}$) at and below 200°C .³⁷ The present results thus provide strong evidence that Si self-interstitials are not injected during the formation of η' -Cu₃Si. This is contrary to the suggestion by Ronay and Schad³⁴ that the formation enthalpy of Si self-interstitials is drastically lowered by the formation of metal-rich silicides. The formation enthalpy of self-interstitials in silicon is in excess of 5 eV,³⁸ resulting in an almost vanishing equilibrium concentration at 200°C . The present results thus raise doubt regarding the applicability of models,³⁹ which relate the type of point defect injected into silicon to the dominant moving species during silicidation; silicon diffusion leads to vacancy injection, while metal diffusion leads to injection of Si self-interstitials.

V. SUMMARY

We have investigated the interaction between Cu and irradiation-induced defects in Si using Schottky-barrier structures formed by the deposition of Cu on *n*- and *p*-type Si irradiated with 2-MeV electrons at room temperature. An anomalously high annealing rate of the V_2 centers is observed at temperatures below 150°C in both *n*- and *p*-type Si, and is attributed to passivation of their electrical activity by the highly mobile Cu through the formation of neutral complexes. A prominent level located at $E_v + 0.52 \text{ eV}$ appears in *p*-type Si after Cu deposition at room temperature. It is suggested that this level originates from a complex formed through interaction of Cu with the carbon-oxygen centers giving rise to the $E_v + 0.35 \text{ eV}$ level. This latter level, which is normally stable up to $\sim 350^\circ\text{C}$, anneals rapidly at 100°C through the formation of the $E_v + 0.52 \text{ eV}$ level, which disappears upon silicide formation. Furthermore, the present results show that no injection of Si self-interstitials into the silicon takes place during the formation of the metal-rich silicide η' -Cu₃Si.

ACKNOWLEDGMENTS

It is our pleasure to acknowledge D. Mikalsen of the Central Scientific Services Material Laboratory at IBM Yorktown Heights, NY for assistance in preparing the

Schottky-barrier structures. Financial support was received from the Swedish Natural Science Research Council and the Swedish Board for Technical Development (NUTEK).

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