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Annealing behavior of the EB-centers and M-center in low-energy electron irradiated *n*-type 4H-SiC

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After low-energy electron irradiation of epitaxial *n*-type 4H-SiC with a dose of $5 \times 10^{16} \text{ cm}^{-2}$, the bistable M-center, previously reported in high-energy proton implanted 4H-SiC, is detected in the deep level transient spectroscopy (DLTS) spectrum. The annealing behavior of the M-center is confirmed, and an enhanced recombination process is suggested. The annihilation process is coincidental with the evolution of the bistable EB-centers in the low temperature range of the DLTS spectrum. The annealing energy of the M-center is similar to the generation energy of the EB-centers, thus partial transformation of the M-center to the EB-centers is suggested. The EB-centers completely disappeared after annealing temperatures higher than 700 °C without the formation of new defects in the observed DLTS scanning range. The threshold energy for moving Si atom in SiC is higher than the applied irradiation energy, and the annihilation temperatures are relatively low, therefore the M-center, EH1 and EH3, as well as the EB-centers are attributed to defects related to the C atom in SiC, most probably to carbon interstitials and their complexes. © 2011 American Institute of Physics. [doi:10.1063/1.3586042]

I. INTRODUCTION

SiC is a promising material for very high bipolar power devices if the carrier lifetime can be increased and the lifetime limiting defects can be identified. Studies by Hiyoshi and Kimoto^{1,2} detected the responsible defect level $Z_{1/2}$ and EH6/7. After thermal oxidation, these defects were eliminated and the carrier lifetime significantly increased. Also the carbon-implantation/annealing process reduced the defect concentration of $Z_{1/2}$ as reported by Storasta *et al.*³ Both studies give strong indications that $Z_{1/2}$ and EH6/7 should be related to the carbon vacancy. These two levels are already present in as-grown 4H-SiC epitaxial layers.

During device processing, e.g., implantation or irradiation steps, additional defects can be introduced, which can limit the device performance. Intentional irradiation is used to study the resulting defect levels in the bandgap. Deep level transient spectroscopy (DLTS) is a good choice for obtaining information about the energy position of the introduced defects in the bandgap as well as for determining the defect concentrations.

High-energy irradiation in the range of MeV introduces Si and C vacancies and their complexes in SiC. DLTS investigations revealed the irradiation induced defects, such as EH1, EH3, EH4, and EH5.⁴ The S-center, which was detected in proton-implanted (2.5/2.9 MeV) and in electron irradiated (15 MeV) *n*-type 4H-SiC material by David *et al.*,⁵ gives rise to DLTS peaks S_1 and S_2 , which are likely the same as the

observed EH1 and EH3. Both peaks have a one-to-one ratio and are assigned to the same defect of different charge states. The annealing of the S-center follows a first order process and takes place above 600 K with an activation energy of $E_a = 1.8 \text{ eV}$ and a frequency factor of $\nu \approx 1 \times 10^{11} \text{ s}^{-1}$. The DLTS peaks, labeled S_2 and S_4 , detected in 8.2 MeV irradiated *n*-type 4H-SiC^{6,7} showed similar behavior. The origin of these peaks were attributed to a highly mobile defect, recombining at rather low temperatures. In 2 MeV proton-implanted *n*-type 4H-SiC, a bistable defect, the M-center,⁸ was observed. The defect has two configurations A and B. Its annealing behavior was further investigated in Refs. 9 and 10 and found to follow a first order kinetics with $E_a = 2.0 \text{ eV}$ and a prefactor of $R_0 = 1.5 \times 10^{13} \text{ s}^{-1}$, which was associated with a pure dissociation process.

By using low-energy irradiation (<220 keV), carbon interstitials and vacancies, as well as their complexes, can be studied because the energy is not enough to move the Si in the SiC lattice.¹¹ Several DLTS studies of low-energy irradiated *n*-type 4H-SiC exhibit the above discussed EH1 and EH3 levels^{12–14} (labeled ET1 and ET2 by Danno *et al.*¹⁴), which were attributed to carbon related defects. Storasta *et al.*¹² showed that the EH1 and EH3 peaks disappear simultaneously after annealing at about 650 K without the introduction of new peaks in the DLTS spectrum.

In contrast to the previous statement, we have shown that bistable defects, called EB-centers, evolved¹⁵ after annihilation of EH1 and EH3. The transformation between two configurations, labeled I and II, takes place at room temperature depending on if a bias is applied to the sample ($II \rightarrow I$) or not ($I \rightarrow II$).

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Annealing can recover irradiation induced defects of which some are highly mobile⁵ and move already at low annealing temperatures, e.g., the annihilation of EH1 and EH3 is proposed to be caused by the recombination of thermally unstable C_i ^{6,7,12,14} or by the dissociation of a complex defect.¹⁶ Calculations show the thermal instability of the mobile C_i .^{17–20} But if recombination of interstitials takes place, the concentration of their counterparts, vacancies, should decrease as well.¹² Several studies in different temperature ranges and for different irradiation energies were previously presented. Multistage annealing processes are calculated for Frenkel pairs in irradiated SiC²⁰ and measured in electron irradiated *n*-type 4H-SiC;¹⁶ highly movable related defects anneal first before elementary, stable defects start to decrease, e.g., $Z_{1/2}$ and EH6/7 above 1700 °C.^{12,16,21,22}

The annealing can be enhanced under injection of carriers due to electron-hole recombination at the defect site,²³ which may change the charge state.²⁴ The defect migration is thus increased.²⁵ Recombination enhanced annealing was previously reported for the EH1 and EH3 peaks in low-energy electron irradiated 4H-SiC.²⁶

In this study, low-electron irradiated *n*-type 4H-SiC epilayers were investigated by DLTS. The bistable M-center was observed, and its annealing behavior under different bias conditions was studied. During the annihilation process, the above mentioned bistable EB-centers evolved. Isochronal annealing between 480 and 900 °C revealed the annealing process of the EB-centers.

II. EXPERIMENTAL DETAILS

Using homo-epitaxially chloride based chemical vapor deposition, a 200 μm thick low doped *n*-type layer was grown on a highly doped *n*-type 4 H-SiC substrate.²⁷ Low-energy (200 keV) electron irradiation was performed at room temperature on parts of the samples with a dose of $5 \times 10^{16} \text{ cm}^{-2}$. Ni-Schottky contacts with a thickness of

around 1000 Å were deposited by thermal evaporation on to the front side of the as-grown and the irradiated samples. Conducting silver paint served as an ohmic backside contact to the highly doped substrate. Capacitance voltage measurements at room temperature revealed a homogenous doping with a net concentration of $N_d - N_a \approx 1 \times 10^{15} \text{ cm}^{-3}$. DLTS spectra were taken in the temperature range between 85 and 700 K. The DLTS parameters were: filling pulse length of 10 ms, filling pulse height of 10 V, and reverse bias of $V_r = -10 \text{ V}$. A boxcar technique was used for the evaluation of the capacitance transients. In some cases, lock-in amplification simulation resulted in improved signal to noise ratio. Isothermal ($T = \text{constant}$) as well as isochronal ($t = \text{constant}$) temperature treatments were performed directly in the cryostat for annealing temperatures less than 750 K with the ability to control the bias conditions during annealing. For higher temperatures ($T_{\text{anneal}} = 480 - 900 \text{ °C}$), a furnace under Ar-ambient was used. An additional high temperature annealing ($T_{\text{anneal}} = 1600 \text{ °C}$) was achieved using a sublimation growth reactor under vacuum conditions.

III. RESULTS AND DISCUSSION

A. The M-center and the EB-centers in low-energy electron irradiated 4H-SiC

In Fig. 1(a), a typical DLTS spectra for the as-grown sample and after low-energy electron irradiation is shown. The DLTS amplitude of the $Z_{1/2}$, which is the only defect present in the as-grown DLTS spectrum for temperatures lower 400 K, is increased by irradiation as previously reported by Danno *et al.*¹⁴ After irradiation, the EH1 and EH3 (labeled ET1 and ET2,¹⁴ S1 and S2,⁵ or S2, S4^{28,29}) are observed. The bistable M-center with its two configurations A and B is detected. As discussed by Martin *et al.*^{8,9} and as can be seen from Fig. 1, the M-center is superimposing the EH1, $Z_{1/2}$, and EH3 peaks. Depending on the bias applied to the sample during cooling prior to DLTS measurement, the

FIG. 1. (Color online) (a) Typical DLTS spectra for as-grown material (dotted line) and after low-energy electron irradiation (circles and squares). Irradiation induced defects, EH1 and EH3, are shown as well as the bistable M-center in configuration A (circles) and configuration B (squares). Inset: Difference in DLTS signals between the A and B. (b) Low temperature DLTS spectrum showing the EB-peaks giving rise to configurations I (filled circles) and II (open squares). DLTS parameters: rate window $(3.2 \text{ s})^{-1}$, $t_p = 10 \text{ ms}$ and $V_r = -10 \text{ V}$.

TABLE I. Properties of the M-peaks: energies and capture cross sections are obtained from Arrhenius plots ($\ln(e/T^2)$ versus $1000/T$) and concentrations from the DLTS peak amplitudes.

Config.	peak	E_a (eV)	σ (cm ²)	N_t (cm ⁻³)
A	M1	$E_C - 0.41$	5×10^{-15}	6.1×10^{13}
A	M3	$E_C - \approx 0.9$	5×10^{-13}	5.6×10^{13}
B	M2	$E_C - 0.70$	7×10^{-14}	5.8×10^{13}

spectrum changes. Cooled from room temperature with applied bias, configuration A, which gives rise to DLTS peaks M1 and M3, is observed. The defects are only detected in configuration B (DLTS peak M2) after cooling from about 450 K without bias applied to the sample as seen as squared symbols in Fig. 1. The activation energies as well as the capture cross sections for the M-peaks are in good agreement with the peaks observed after high-energy proton-implantation,¹⁰ see Table I.

The inset of Fig. 1 shows the difference in DLTS signal between the two configurations. The amplitudes are similar for M1 and M2 and slightly less for M3 because M2 and M3 are overlapping and thus partly cancel out during subtraction. Martin *et al.*⁸ and Nielsen *et al.*,¹⁰ attributed M1 and M3 to the same defect configuration but different charge states and M2 to another defect configuration. Because the M-center is detected after low-energy irradiation, it may be attributed to carbon related defects. Gali *et al.*¹⁹ calculated the activation energies of C_i and complex metastable C_i ring structures, which have levels in the range of those of the M-center, see Table I.

In Fig. 1(b), the DLTS spectra of the two configurations I and II of the EB-peaks are shown. The peaks EB4 and EB5/6 are present if a bias is applied during cooling from room temperature prior to DLTS measurement. If the sample is cooled without bias instead, peaks EB1, EB2, and EB3 occur in the DLTS scan. The bistable behavior of the EB-centers is

investigated and discussed in detail elsewhere¹⁵ as well as as-signation to carbon related complex defects.

B. Annealing of M-center and generation of EB-centers

Nielsen *et al.*⁹ studied the annealing behavior of the M-center. The annihilation process follows first order kinetics according to:

$$R = R_0 \exp\left(\frac{-E_a}{k_B T}\right) \quad (1)$$

with a thermal activation energy of $E_a = 2.0$ eV and a pre-factor of $R_0 = 1.5 \times 10^{13} \text{ s}^{-1}$, which is in good agreement with the lattice vibration frequency for 4H-SiC at 300 °C,³⁰ and thus the annealing out was assigned to a defect dissociation process.¹⁰

Contrary to Nielsen *et al.*,¹⁰ who measured the sample twice after each annealing step to obtain information on both configurations, and thus having the possibility to subtract the two spectra, we conducted the isochronal annealing on two different samples. This gave us the possibility to observe possible recombination enhanced defect reactions and/or the formation of new defect levels depending on the bias condition during annealing. In Fig. 2, the results from the isochronal annealing ($t_{\text{anneal}} = 5$ min) are shown for different bias conditions in the low-energy electron irradiated material. In the case when the sample was annealed with bias (filled symbols in Figs. 2(a) and 2(b)), the M-center was stabilized in configuration A during the heat treatment and the subsequent measurement. The defect concentration of the $Z_{1/2}$ level, which was obtained from the DLTS peak height, stays in the low 10^{14} cm^{-3} range, seen in linear scale in Fig. 2(b), while the peak amplitudes representing the overlapping EH1 and M1 as well as EH3 and M3 are gradually decreasing for temperatures higher than 400 K, displayed in logarithmic scale in Fig. 2(a). This gradual change can be explained by a smaller depletion width at higher temperatures and thus a

FIG. 2. (Color online) Isochronal annealing study, (a) (logarithmic concentration scale) and (b) (linear concentration scale) annihilation process of the M-center, EH1 and EH3 observed with ($V_r = -10$ V, filled symbols) and without bias ($V_r = 0$ V, open symbols) during annealing and cooling. (c) Generation process of the EB-centers; EB-peaks in the low temperature region of the DLTS spectra, studied with (filled symbols) and without (open symbols) applied electric field during heat treatment and followed cooling period. Solid lines are only guidelines for the eyes.

partial transformation to configuration B. All peaks, besides the $Z_{1/2}$, are completely annealed out at 700 K. Coincidental to the annihilation process, the EB-centers evolve in configuration I, consisting of peaks EB4 and EB5/6 (filled symbols in Fig. 2(c)), in the low temperature part of the DLTS spectrum. The appearance of the EB-peaks is in contrast to most previous reports claiming that no peaks were detected during and after the disappearance of EH1 and EH3.^{5,12} Storasta *et al.*¹² mentioned that the defects are either completely annihilated or transferred to an electrically inactive defect. In high-energy (8.2 MeV) electron irradiated 4H-SiC, Castaldini *et al.*²⁹ observed changes in the DLTS spectrum after annealing at 573 K; S2 and S4 similar to EH1 and EH3, respectively, completely disappeared, while the two peaks S1 and S5 split into several components after annealing. These S1 A and S1B may be similar to EB5/6, but their possible metastability was not investigated. Castaldini attributed these defects to complexes with high formation energy. The defect concentrations of the new peaks EB4 and EB5/6 are in the mid range of 10^{12} cm^{-3} , one order of magnitude less than those of the M-center, EH1 and EH3.

The same isochronal annealing procedure was done without applied bias as displayed in Figs. 2(a) and 2(b) with open symbols. Up to an annealing temperature of about 400 K, the defect concentrations are the same as for the case of applied bias. This can be explained by the fact that the M-center is still in configuration A. Between 400 and 450 K, the M-center is completely transferred to configuration B as discussed in the preceding text. The transition to configuration B can also be seen by an increase of the $Z_{1/2}$ peak amplitude, which actually now represents the sum of $Z_{1/2}$ and M2, seen in Fig. 2(b). The annihilation process of EH1 and EH3, which can now be observed independently of M1 and M3, seen in Fig. 2(a) starts at about 600 K and is completed at about 650 K, i.e., at lower temperatures than the annealing with an electric field applied. This behavior may indicate a recombination enhanced process, which was shown to be present for the EH1 and EH3 peaks by Storasta *et al.*¹³ The concentration of the M-center, now observable as M2 overlapping the $Z_{1/2}$ starts to decrease at lower temperatures than the M1 and M3, thus also here an enhanced annealing may take place. At 625 K, the EB-centers in configuration II, giving rise to peaks EB1, EB2, and EB3 (open symbols in Fig. 2(c)), appear in the DLTS spectrum, which are different from EB4 and EB5/6.

Because the annealing process of the M-center, as well as EH1 and EH3 occurs at relatively low temperatures, the origin of these defects may be related to mobile species, e.g., carbon interstitials, C_i or their complexes. Additionally, the defects are present after low-energy electron irradiation, which suggests carbon related defects because the threshold to move the silicon atom in SiC during irradiation is not reached.

To determine the activation energy for the formation of the EB-centers, see Fig. 3(b), we choose the EB5/6 peak, which showed the highest DLTS amplitude for the generation investigation. An isothermal annealing study under applied bias was performed at six distinct temperatures. The annealing time was increased until the DLTS amplitude of

FIG. 3. (Color online) (a) EB5/6 generation rate for $T = 590 \text{ K}$. (b) Isothermal annealing at $T = 550 \text{ K}$, 560 K , 570 K , 590 K , 600 K , and 610 K without applied electric field during heat treatment and followed cooling period. Arrhenius plot of generation rates of the measured EB-centers (circles) and of the simulated M-center (squares).

the EB5/6-peak, under consideration, was saturated and thus the saturation defect concentration N_T^∞ was estimated, see Fig. 3(a). Knowing N_T^∞ , the generation rates R were determined from the exponential fitting:

$$N_T(t) = N_T^\infty (1 - \exp(-Rt)) \quad (2)$$

The thermal activation energy for the formation of the EB-centers, E_a can then be calculated from the determined generation rates using Eq. 1.

In Fig. 3(b), $\ln(R)$ versus $1/(k_B T)$ is displayed. By fitting we determined $E_a = 2.0 \pm 0.5 \text{ eV}$ and $R_0 = 4 \times 10^{14} \text{ s}^{-1}$. Because the DLTS signal of the EB-peaks is close to the detection limit of our setup, the exact determination of the defect concentration is difficult and thus the calculation of the generation energy using the generation rates resulted in $\pm 0.5 \text{ eV}$ error range. For four temperatures, the annihilation process of the M-center was simulated using the results for the annihilation energy and the prefactor given in Ref. 9. The obtained EB-centers generation energy is close to the annihilation energy of the M-center, thus these two kinds of centers are likely correlated. The prefactor will be even more affected by error, thus one order of magnitude more or less can be assumed. The measured $R_0(\text{EB-centers})$ is thus comparable to $R_0(\text{M-center})$, which was in the range of the calculated vibration frequency of SiC³⁰ at 300°C , $\nu = 1.6 \times 10^{13} \text{ s}^{-1}$ and which is close to the prefactor typical for atomic jump processes.³¹ The DLTS amplitudes of the M-center and thus the defect concentration is much higher than the one of the EB-centers, thus only a small amount of the M-center may be transferred to the EB-centers. This may

FIG. 4. (Color online) (a) DLTS spectra for different annealing temperatures and different bias conditions prior to DLTS measurements. DLTS parameters: rate window $(4.7 \text{ s})^{-1}$, $t_p = 10 \text{ ms}$ and $V_r = -10 \text{ V}$. (b) EB5/6 defect concentration after isochronal annealing for $t = 30 \text{ min}$ at different temperatures. The solid line is only a guideline for the eyes.

be explained by competing processes, e.g., recombination of C_i with V_C and the formation of C_i -aggregates as discussed by Bockstedte *et al.*¹⁸ using *ab initio* calculations.

C. Annealing of EB-centers

In a final step, the annealing behavior of the generated EB-centers, which was not discussed in our previous study,¹⁵ was investigated. Therefore, the samples were annealed for $t_{\text{anneal}} = 30 \text{ min}$ in a furnace under Ar atmosphere. Afterward new contacts of different diameter size were evaporated on the samples. Figure 4(a) shows the DLTS spectra for different annealing temperatures. For the 500 and 600 °C heat treatments, the EB-centers were still present in both configurations. However, an additional peak, labeled EB7 occurred after annealing at ($T = 500 \text{ °C}$) with an activation energy of $E_a = E_C - 0.29 \text{ eV}$. The EB7 peak did not show any metastable behavior caused by the applied bias. After annealing at 700 °C and higher, all EB-peaks annealed out. Figure 4(b) shows the decreasing defect concentration of the EB5/6 peak with increasing annealing temperature. At about 700 °C, the defect completely anneals out without the formation of new peaks in the investigated DLTS temperature range. One sample was further annealed in a sublimation reactor at 1600 °C. Only slightly reduced signatures of $Z_{1/2}$ or EH6/7 are left in the DLTS spectrum as previously reported by several authors.^{16,21,22} The annealing of these two centers takes place at rather high temperatures, $T \approx 2000 \text{ °C}$.

The annealing of the EB-centers occurs at higher temperatures than for the M-center and the EH1/EH3, which

were proposed to be due to the recombination of mobile carbon interstitials but at lower temperatures than the $Z_{1/2}$ or EH6/7, which were attributed to the carbon vacancy. The EB-centers are thus likely carbon interstitial related complex defect.

IV. CONCLUSION

Low-energy electron irradiated *n*-type 4H-SiC was investigated. The bistable M-center was observed, and its annealing behavior was confirmed. During the annealing, which was electric field dependent and possibly caused by an enhanced recombination defect process, the bistable EB-centers evolved. The annihilation energy of the M-center is in agreement with the generation energy of the EB-centers, thus during annealing parts of the M-center transform into the EB-centers. At the same time, the irradiation induced peaks EH1 and EH3 annihilate simultaneously. The annealing takes place at low temperatures, thus a mobile species, like C_i , and their complexes have to play an important role. The EB-centers, themselves annihilate at about 700 °C without the generation of new defects.

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