Electrical characterization of electron irradiated and annealed lowly-doped 4*H*-SiC

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Abstract

The effect of high energy electron (HEE) irradiation on nickel Schottky contacts fabricated on lowly-doped *n*-type 4*H*-SiC was investigated by deep level transient spectroscopy (DLTS) and high resolution Laplace-DLTS. The Schottky contacts were deposited by resistive evaporation of nickel and were observed to be of good rectification quality from current-voltage measurements. DLTS was performed up to 350 K to investigate the presence of defects before and after HEE irradiation. HEE irradiation was observed to induce three deep level defects below 350 K at 0.42 eV, 0.62 eV and 0.76 eV below the conduction band minimum. These deep level defects are labelled $E_{0.42}$, $E_{0.62}$ and $E_{0.76}$. Defects $E_{0.42}$ and $E_{0.76}$ were observed after the same electron fluence and were annealed out at the same temperature, suggesting that the defects could be strongly related. The effect of HEE irradiation and annealing on as-grown defects was also investigated and is reported.

Keywords: 4*H*-SiC, Schottky contacts, high energy electron irradiation, DLTS

1. Introduction

Silicon carbide is a wide bandgap semiconductor with desirable physical and electronic properties that makes it suitable for fabrication of high frequency, high temperature and high power devices [1-3]. SiC is also a radiation hard material making it a material of choice for devices that can operate in radiation harsh environments and at high temperatures [4]. Such devices include radiation detectors and devices for space applications. As with other

semiconducting materials, use of a semiconductor in electronic device fabrication depends on the knowledge of presence of deep levels. This is specially so since defects can either enhance or degrade the devices. Thus, if the presence and effect of defects is well understood, controlled introduction into the material can be performed to enhance performance or the defects can be removed either by annealing or by other methods.

One way of controlled introduction of defects in semiconductors is by particle irradiation. This can be done by varying particle energy and fluence. Irradiation induces vacancies and interstitials in semiconductors, although many other complex defects can also result. Complex defects resulting from particle irradiation include Frenkel pairs, antisites (such as in compound semiconductors), vacancy pairs, etc. Particle irradiation is also used to shed light on the origin and in some instances on the microstructural nature of defects. In SiC for example, low energy particle irradiation can be used to differentiate between carbon and silicon related defects since these elements have a different threshold displacement energy. Irradiation with particles with low energy that is insufficient to produce silicon vacancies or related defects can thus only induce carbon vacancies and related defects.

Defects are described majorly using two parameters; the defect activation energy and the defect capture cross section. The defect activation energy, E_T, refers to the position of the defect level with respect to either the conduction band or the valence band. The capture cross section, σ_n, relates to how effective the defect is in trapping free carriers. The defect concentration is also another parameter used to quantify a defect level.

To the best of our knowledge, the characterization of low doping density (~4×10^{14} cm^{-3}) 4H-SiC after electron irradiation and thermal annealing has not been studied by high-resolution Laplace deep level transient spectroscopy (Laplace-DLTS). In this study, high energy electron (HEE) irradiation was performed using a strontium source on nitrogen doped 4H-SiC with a doping density of ~4×10^{14} cm^{-3}. Defects present in as-prepared as well as defects introduced

by irradiation were characterized by conventional DLTS and Laplace-DLTS. Laplace-DLTS was used to separate defect levels with closely spaced emission rate. Annealing studies were also performed to study the annealing kinetics of the irradiation induced-defects as well as the as-prepared and process-induced defects.

2. Experimental procedure

The samples used in this work were epitaxially grown, N-doped, n-type 4H-SiC wafers supplied by CREE Research Inc. The epilayer was grown by chemical vapour deposition on the Si-face of the SiC substrate, which has a low net doping density of ~4.0×10^{14} cm^{-3}. The orientation and thickness of the samples are 8.05° and 19.20 microns, respectively. Samples were degreased by boiling in trichloroethylene, acetone and methanol for 5 minutes each. This was followed by rinsing in de-ionised water before a 30 seconds dip in hydrofluoric acid to remove the native oxide layer. Samples were then rinsed in de-ionised water followed by blow drying using N$_2$ gas. Nickel ohmic contact with a thickness of 300 nm was deposited by joule evaporation onto the highly doped (1×10^{18} cm^{-3}) side of the sample and annealed in flowing argon for 10 minutes at 950 ˚C. Prior to Schottky contact fabrication, the same cleaning procedure was repeated except that instead of boiling, 3 minutes rinsing in each of the three solvents was performed in an ultrasonic bath. Nickel Schottky contacts with a diameter of ~0.6 mm and a thickness of 100 nm were then resistively deposited onto the lowly doped side of the samples. DLTS measurements were carried out using a National Instruments Digital Acquisition (DAQ) based Laplace-DLTS system [5]. HEE irradiation of the diodes was performed using a Sr-90 radioactive source. Strontium decays with an emission of ~0.55 MeV into yttrium which then decays zirconium with an emission of ~2.3 MeV. The electrons emitted from the strontium source thus have a continuous energy distribution, with more than 70 % having more than 0.25 MeV. Detailed information on the irradiation source can be obtained from Auret *et*. *al*. [6]. The diodes were annealed after irradiation in steps of 100 ˚C in flowing argon gas up to 600 ˚C.

3. Results and Discussion

Current–voltage (*I–V*) and capacitance–voltage (*C–V*) measurements were carried out after Schottky contacts deposition to determine the rectification properties of the prepared Schottky contacts. The contacts had excellent rectification properties making it possible for DLTS measurements to be carried out on the samples. *I–V* and *C–V* measurements were also repeated after each irradiation step to determine if the diodes were still suitable for DLTS measurements. Parameters which were monitored from these measurements to determine the suitability of the diode for DLTS measurements include the ideality factor (*n*), the Schottky barrier height obtained from both *I–V* (Φ_{I-V}) and *C–V* (Φ_{C-V}) and the reverse leakage current measured at −5 V. The ideality factor was obtained assuming the thermionic emission process to be the dominant current transport mechanism across the Schottky barrier. A summary of the obtained *I–V* and *C–V* measurements is presented in Table 1, as earlier reported in *ref*. [7]. A more detailed *I–V* and *C–V* study on characteristics of 4H-SiC with HEE irradiation can be obtained from Omotoso *et al*. [8] It was demonstrated that 4H-SiC retains its rectification properties after fluence of 6×10^{14} cm^{-2} at room temperature [8].

Table 1. The diode ideality factor, Schottky barrier height and reverse leakage current obtained before irradiation, after irradiation and after annealing the irradiated diodes at 600 °C.

Fluence	$n \pm 0.02$	Φ_{I-V} (eV) ± 0.02	Φ_{C-V} (eV) ± 0.02	I_L (A) at −5V
0	1.05	1.62	2.13	1.2×10^{-14}
5.4×10^{14} cm^{-2}	1.04	1.60	2.14	5.2×10^{-13}
5.4×10^{14} cm^{-2} + 600 ºC annealing	1.06	1.58	2.20	3.2×10^{-12}

DLTS was carried out on the diodes before HEE irradiation, after HEE irradiation and during annealing of the irradiated diodes. A control measurement was done by annealing as-prepared diodes and taking DLTS measurements. Annealing the as-prepared diodes between 100 °C and 700 °C did not induce new defects. Any defect that may be observed during annealing of irradiated diodes is therefore as a result of irradiation and not annealing.

3.1 Irradiation results

DLTS measurements were carried out in the temperature range 30 K $\leq$ T $\leq$ 350 K. The reverse bias was maintained as 5 V while a forward voltage pulse of -1 V with a pulse width of 1 ms was applied. Normalized DLTS spectra obtained using a 2.5 s^{-1} rate window is given in Fig. 1 showing the presence of four defect levels. The energy levels were labelled $E_{0.10}$, $E_{0.12}$, $E_{0.18}$ and $E_{0.69}$ with activation energies of $E_C - 0.10$ eV, $E_C - 0.12$ eV, $E_C - 0.18$ eV and $E_C - 0.69$ eV. The level $E_{0.10}$ is a shallow nitrogen donor occupying a cubic lattice site [9, 10]. When occupying a hexagonal site, nitrogen dopants introduce shallow energy levels with an activation energy of ~ 0.055 eV [11, 12].
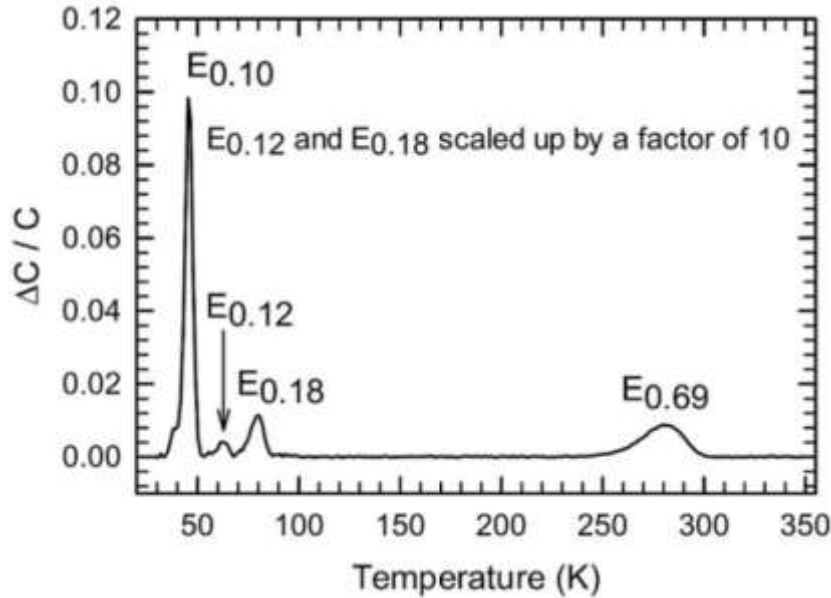

Fig. 1. DLTS spectra for the as-deposited sample. Spectra recorded at 5 V, and rate window of 2.5 s^{-1}.

Energy level $E_{0.12}$ was observed and has been attributed to a titanium impurity [13, 14]. $E_{0.18}$ has also been attributed to a titanium impurity [14, 15]. It is possible that these two defect levels are both induced by a titanium impurity but occupying different geometrical positions in SiC. The defect is seen as $E_{0.12}$ when occupying a hexagonal position while it appears as $E_{0.18}$ when occupying a cubic position. A comprehensive study of the two defect levels is provided by Achtziger *et al*. after implanting 4*H*-SiC with radioactive isotopes of chromium

and vanadium [14]. The authors who also observe defect level $E_{0.18}$ however attribute it to a chromium impurity in contrast to Castaldini *et al* [15]. Metal impurities in as-grown semiconductors are introduced during growth of the semiconductors.

Defect level $E_{0.69}$ observed in this study is widely believed to be of intrinsic nature. From the literature, this defect has been attributed to a carbon vacancy, V_C [16], a silicon vacancy [17], a silicon-carbon divacancy, $V_{Si} + V_C$, a carbon silicon antisite pair, $Si_C + C_{Si}$ [18]. It has also been claimed that the defect could be a silicon antisite–silicon vacancy complex, $C_{Si} - V_{Si}$ [18] and a nitrogen impurity next to a carbon interstitial $I_C + N$ [19]. The problem of whether the defect level is either a silicon or carbon related defect was resolved by Son *et al.*, and has been identified as double acceptor of an isolated carbon vacancy [20].

The normalized DLTS spectra obtained in this study after HEE irradiation are shown in Fig. 2. The presented spectra were obtained using a reverse bias of 5 V, a forward pulse of –1 V with a pulse width of 1 ms at a 2.5 s^{-1} rate window. The *signatures* (activation energy and apparent capture cross section) of the deep level defects show in Fig. 3 were determined as reported earlier [21].
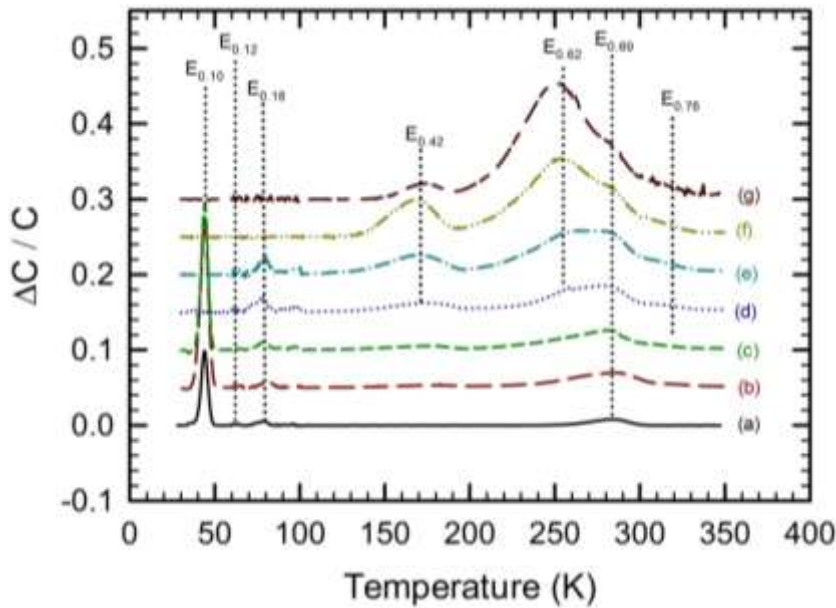

Fig. 2. Spectra produced at different electron fluence irradiations. (a) as-prepared, (b) 9.8×10^{13} cm^{-2}, (c) 2.0×10^{14} cm^{-2} (d) 2.9×10^{14} cm^{-2}, (e) 3.9×10^{14} cm^{-2}, (f) 4.9×10^{14} cm^{-2} (g) 5.4×10^{14} cm^{-2}.
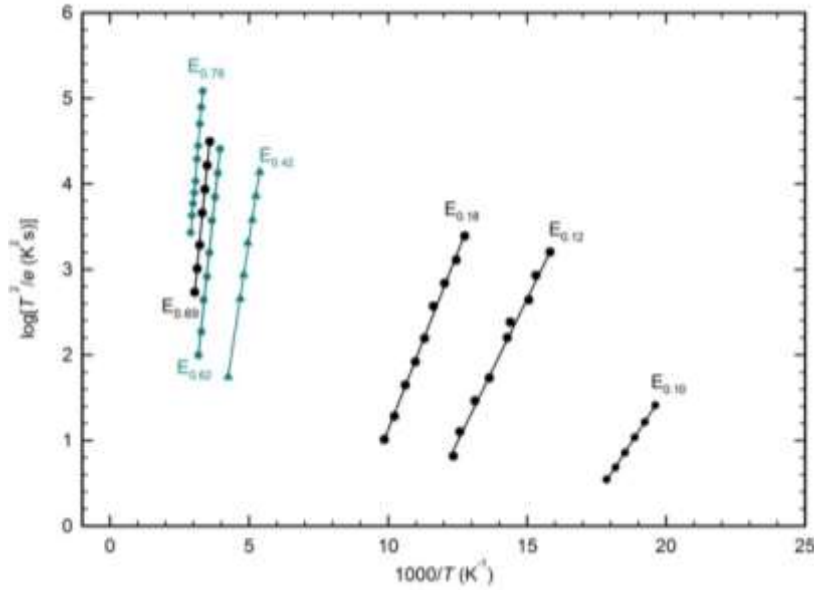
Fig. 3. Arrhenius plots of the defects obtained before and after HEE irradiation.

Using step by step high energy electron irradiation, defect level $E_{0.69}$ was observed to increase in concentration from 2×10^{13} cm^{-3} before irradiation to 9×10^{13} cm^{-3} after a total fluence of 5.4×10^{14} cm^{-2}. Apart from level $E_{0.69}$, another defect level with activation energy of $E_C - 0.62$ eV was observed as the fluence increased. After an electron fluence of 4.9×10^{14} cm^{-2}, the concentration of level $E_{0.62}$ had surpassed that of level $E_{0.69}$, and the level $E_{0.69}$ was only appearing as a shoulder of $E_{0.62}$. The two broad defect levels obtained by conventional DLTS were separated more accurately into three distinct defect level by high resolution Laplace-DLTS as shown in Fig. 4. We have also observed the defect level $E_{0.62}$ after 5.4 MeV alpha-particle irradiation on 4*H*-SiC [22, 23]. In general, it may be deduced that different types of irradiation induced the same type of defects although in different concentrations depending on the energies of the irradiation particles as well as their masses.
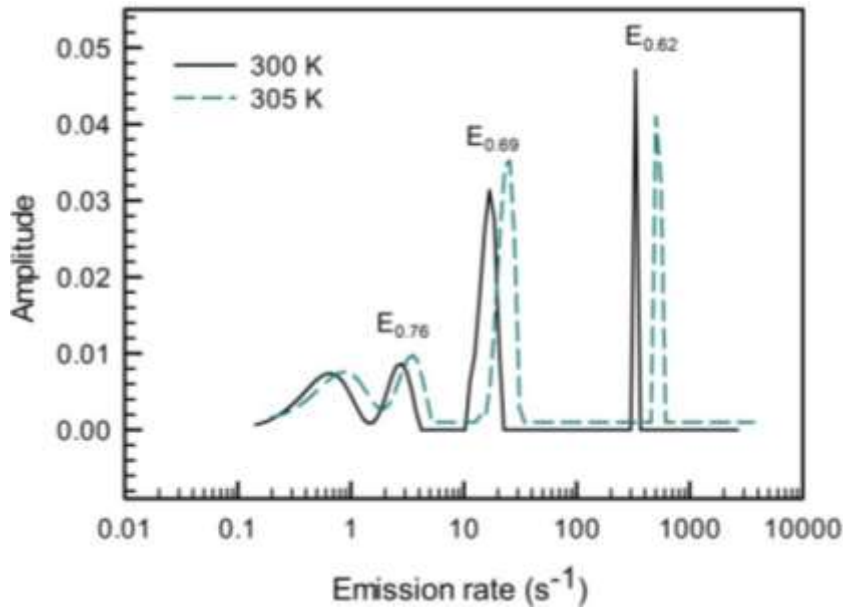
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Fig. 4. Laplace-DLTS spectra showing emission rates of levels $E_{0.62}$, $E_{0.69}$ and $E_{0.76}$. The fourth peak was determined to be a measurement artefact.

Defect level $E_{0.69}$ is commonly referred to as the Z_1/Z_2 defect. This level is believed to be a defect complex consisting of two closely spaced levels Z_1 and Z_2 that have negative U behaviour [24]. After 5.4 MeV alpha particle irradiation of 4*H*-SiC, Ashgar *et al.* [25] proposed the defect exist as a single level but is transformed into a complex by irradiation. This attribute to an introduction of another level, Z_2 which is a carbon vacancy and is superimposed on Z_1. In this study, the irradiation electrons had enough energy to displace carbon from its lattice position hence the attribution of the $E_{0.62}$ level to a carbon vacancy is supported. This does not however rule out the possible participation of carbon in the structure of the defect since the electron energy is enough to create carbon related defects. The displacement energy for carbon in a SiC lattice is approximately 19 eV for the carbon sublattice and 38 eV for the silicon sublattice [26].

After a total fluence of 1.5×10^{14} cm^{-2}, a defect level with energy $E_C - 0.42$ eV was observed with a low concentration of 3.8×10^{12} cm^{-3}. The concentration of the defect increased to 6.2×10^{13} cm^{-3} after a 5.4×10^{14} cm^{-3} fluence. This defect shows acceptor like properties [27, 28] and has been attributed to a silicon vacancy [29]. In addition to $E_{0.42}$ and $E_{0.62}$, another deep

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level is introduced by high energy electron irradiation at $E_C - 0.76$ eV, with a capture cross section of 1.0×10^{-14} cm^2. This defect appeared as a shoulder of $E_{0.69}$ on the high temperature side. The structure of this defect has not been reported in literature despite its presence having been reported after alpha-particle, electron and proton irradiation [27, 30, 31]. However, since this defect is introduced by irradiation, it is possibly generally related to carbon vacancies, silicon vacancies, carbon or silicon interstitials, carbon or silicon antisites or complexes of these possibilities.

The defect energy levels (E_T), apparent capture cross sections (σ_{app}) and the defect concentrations (N_T) determined for each defect are summarized in Table 2.

Table 2. Activation energy E_T, apparent capture cross section σ_{app} and concentration N_T of the observed defects.

Defect label	E_T (eV)	σ_{app} (cm^2)	N_T(cm^{-3})
$E_{0.10}$	0.10 ± 0.01	1×10^{-13}	9.1×10^{13}
$E_{0.12}$	0.12 ± 0.01	7×10^{-15}	2.2×10^{11}
$E_{0.18}$	0.18 ± 0.01	6×10^{-15}	5.4×10^{11}
$E_{0.42}$	0.42 ± 0.03	8×10^{-15}	*6.2×10^{13}
$E_{0.62}$	0.62 ± 0.02	2×10^{-14}	?
$E_{0.69}$	0.69 ± 0.02	8×10^{-15}	*9.3×10^{13}
$E_{0.76}$	0.76 ± 0.03	1×10^{-14}	*4.9×10^{13}

* Defect concentration obtained after electron fluence of up to 5.4×10^{14} cm^{-2}

3.2 Annealing results

After irradiation to a total fluence of 5.4×10^{14} cm^{-2}, the diodes were isochronally annealed in steps of 100 ℃ in flowing argon to a maximum temperature of 600 ℃. The annealing was done at each temperature for 15 minutes and DLTS measurements were repeated after each annealing. The normalized DLTS spectra obtained are shown in Fig. 5. There was no noticeable effect observed after annealing the irradiated samples at 100 °C.
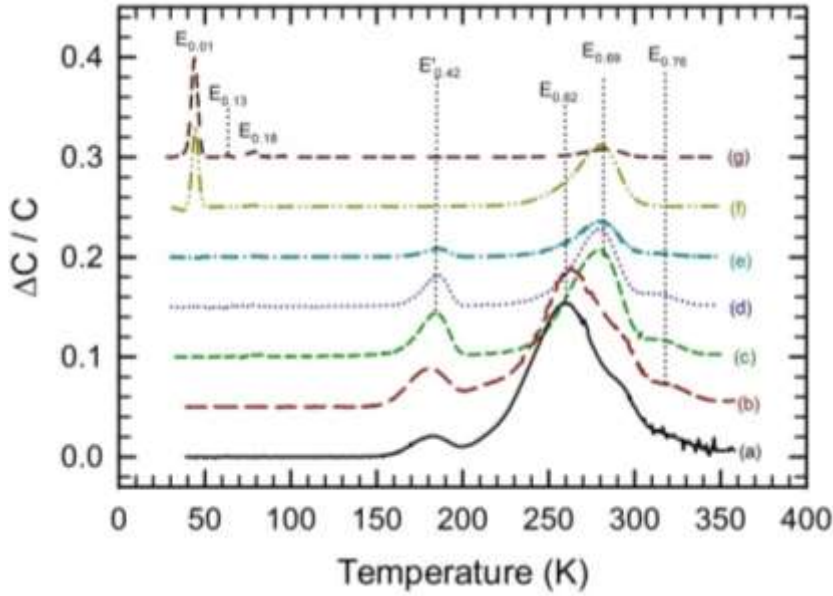
Fig. 5. DLTS spectra obtained after different annealing steps. The spectra obtained before and after irradiation at 5.4×10^{14} cm^{-2} are also shown for comparison. (a) after irradiation, (b) 100 °C, (c) 200 °C, (d) 300 °C, (e) 400 °C, (f) 500 °C and (g) as prepared. The 500 °C and 600 °C spectra were similar hence, the 600 °C spectrum is not shown.

The first annealing induced difference was observed at 200 °C on defect level $E_{0.62}$. The defect was completely annealed out after a 15 minutes heat treatment. The low temperature annealing out of the defect shows that the defect is highly unstable. During the annealing process, the concentration of defect $E_{0.69}$ was observed to decrease systematically. However, after annealing at 600 °C, the concentration of the defect remained above the pre-irradiation concentration levels.

The concentration of both $E_{0.42}$ and $E_{0.76}$ were reduced by annealing. After annealing at 500 °C, both defects were annealed beyond detection levels. Similar annealing dynamics of these defects suggest that either the two defects require the same annealing sink or they act as an annealing sink to the same type of mobile defects [17]. The effect of annealing on levels $E_{0.12}$ and $E_{0.18}$ was not noticeable due to the low concentration of the defects while level $E_{0.10}$ which had been completely suppressed by irradiation showed recovery after annealing at

500 °C. The reason why irradiation suppresses this level with a reversal from annealing has been reported earlier by Omotoso *et al*. [31].

4. Conclusions

High-resolution Laplace DLTS has been successfully used to separate two broad defects (E0.62 and E0.69) obtained by conventional DLTS into three distinct defects.

The high energy electron irradiation up to fluence of 5.4×10^{14} cm^{-2} introduced three defect levels $E_{0.42}$, $E_{0.62}$ and $E_{0.76}$. Laplace DLTS was used to resolve defect level $E_{0.76}$ which partially overlaps with defect $E_{0.69,}$ to give the activation energy with more accuracy. The level $E_{0.62}$ was annealed out after annealing at a temperature of 200 °C. Levels $E_{0.42}$ and $E_{0.76}$ were relatively stable up to annealing temperatures of 400 °C although the concentrations were reduced progressively. After annealing at 500 °C, these levels were completely annealed out. These two defects are proposed to be closely linked in structure due to similar introduction and annealing dynamics. Level $E_{0.10}$ which is a nitrogen impurity was completely suppressed by irradiation but was recovered after annealing at 500 °C.

5. Acknowledgements

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