

# Primary photoluminescence in as-neutron (electron) -irradiated *n*-type 6H-SiC

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Low-temperature photoluminescence spectroscopy has revealed a series of features labeled  $S_1$ ,  $S_2$ ,  $S_3$  in *n*-type 6H-SiC after neutron and electron irradiation. Thermal annealing studies showed that the defects  $S_1$ ,  $S_2$ ,  $S_3$  disappeared at 500 °C. However, the well-known  $D_1$  center was only detected for annealing temperatures over 700 °C. This experimental observation not only indicated that the defects  $S_1$ ,  $S_2$ ,  $S_3$  were a set of primary defects and the  $D_1$  center was a kind of secondary defect, but also showed that the  $D_1$  center and the  $E_1$ ,  $E_2$  observed using deep level transient spectroscopy might not be the same type of defects arising from the same physical origin. © 2006 American Institute of Physics. [DOI: 10.1063/1.2195014]

## I. INTRODUCTION

Silicon carbide (SiC) is a promising wide band gap material for fabricating high-temperature, high-power, high-frequency, and irradiation hard semiconductor devices. Ion implantation is the only available method to realize selective doping in SiC. Unfortunately, some residual defects caused by ion implantation remain even after high-temperature annealing. Further, modern SiC devices are used very widely in radiation environments. All these aspects have motivated researchers to study defects induced by irradiation.<sup>1-5,7-10</sup> Many irradiation-induced deep level defects in 6H-SiC have been reported, among which the most well-known defects are the  $E_1$ ,  $E_2$  observed in deep level transient spectroscopy<sup>1,5-8</sup> (DLTS) and the  $D_1$  center (or  $L_1$ ,  $L_2$ ,  $L_3$ ) as the main signals detected by low-temperature photoluminescence (LTPL) spectroscopy.<sup>9,10</sup> Because of their similar high thermal-stability properties, some investigators have attributed these two groups of defects to the same origin, namely the divacancy ( $V_C-V_{Si}$ ).<sup>1,2,9,11</sup> In our previous work in which defects in *n*-type 6H-SiC samples induced by He implantation and electron irradiation were studied using DLTS techniques, we demonstrated that different particle irradiation indeed introduced different defects.<sup>8</sup>

## II. EXPERIMENTAL

The *n*-type 6H-SiC samples with orientation (0001) used in this work were commercially available from CREE Research, Inc. For these samples, a nitrogen doped epilayer 5 μm thick was grown on *n*<sup>+</sup>-type 6H-SiC substrate. The nitrogen donor concentrations were  $9 \times 10^{15} \text{ cm}^{-3}$  and  $6.6$

$\times 10^{18} \text{ cm}^{-3}$  in the epilayer and the substrate, respectively. Three pieces of samples were used in this work, which were unirradiated, electron irradiated with electron energy of 1.7 MeV, and neutron irradiated, respectively. The two irradiation samples were irradiated with slow neutrons and electrons at room temperature to a dosage of  $1.0 \times 10^{15} \text{ n/cm}^2$  and  $4.5 \times 10^{15} \text{ e/cm}^2$ , respectively. Isochronal thermal annealing was performed in a nitrogen atmosphere between 350 °C and 1100 °C for 30 min. The irradiated samples were stored at room temperature about two weeks before LTPL measurements.

LTPL measurements were performed at 3.5 K and 6 K using a 325 nm wavelength He–Cd laser. The He–Cd laser light was continuous wave and was linearly polarized with respect to the sample. The sample was mounted on the cold finger of a Janis closed cycle cryostat and was illuminated by the laser light, and the incident angle was smaller than 30°. The diameter of laser spot on the sample was estimated to be around 0.1 mm. The laser power was adjusted from 0.5 to 16.8 mW for the variable excitation intensity PL measurement. The LTPL emission was analyzed with a SPEX750M single grating (1200 lines/mm) monochromator and was detected with a Hamamatsu R928 photomultiplier. The lock-in amplifier technology was employed in the setup for optimizing signal-to-noise ratio. The spectral resolution of the spectrometer was 0.1 nm.

## III. RESULTS AND DISCUSSION

A typical PL spectrum of an as-neutron-irradiated *n*-type 6H-SiC sample is shown in Fig. 1(b). Three sets of stable dominant signals were clearly observed. One set of dominant signals is the lines of the well-known 4N0 and the other is the lines labeled as  $S_1$ ,  $S_2$ , and  $S_3$ . The former is related to

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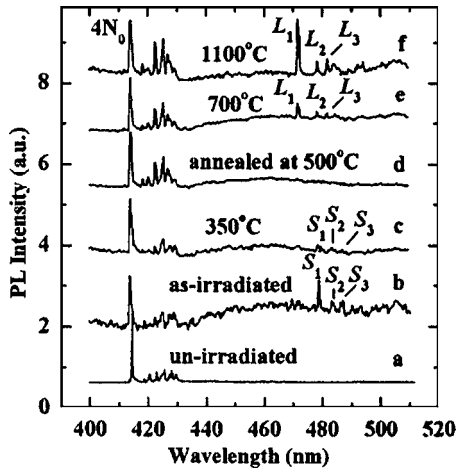


FIG. 1. LTPL spectra for *n*-type 6H-SiC: (a) unirradiated sample at 6 K; and (b)–(f) neutron-irradiated sample at 3.5 K before and after annealing.

bound exciton recombination at a four-particle neutral nitrogen donor at the three inequivalent lattice sites, which has been intensively investigated and clearly identified in Ref. 12 and is commonly used as an indicator for the presence of N dopants in SiC.<sup>13</sup> The LTPL emission lines  $S_1$ ,  $S_2$ ,  $S_3$  at 478.6 nm, 483.3 nm, 486.1 nm, respectively, were observed for the first time. These lines were not seen in the unirradiated sample in this range [Fig. 1(a)] and are very similar to the well-known  $D_1$  center. Similar PL spectra were obtained for the electron-irradiated *n*-type 6H-SiC as shown in Fig. 2.

The annealing behaviors of the irradiated samples were systematically studied as shown in Figs. 1(c)–(f). After annealing at 350 °C, the defect lines  $S_1$ ,  $S_2$ ,  $S_3$  became weak and completely disappeared at 500 °C. Another set of PL peaks emerged at 472.4 nm, 476.9 nm, 482.5 nm after a higher temperature 700 °C annealing. These lines ( $L_1$ ,  $L_2$ , and  $L_3$ ) are the well-known  $D_1$  center, which can withstand annealing up to 1600 °C.<sup>11,13,14</sup> It can be seen that the  $S_1$ ,  $S_2$ ,  $S_3$  and the  $D_1$  center are different since the PL lines of the latter located at 472.4 nm, 476.9 nm, 482.5 nm (corresponding to the three inequivalent lattice sites<sup>15</sup> in 6H-SiC) only emerged after annealing at 700 °C. It is thus most likely that the  $S_1$ ,  $S_2$ ,  $S_3$  is a set of primary defects while the  $D_1$  center

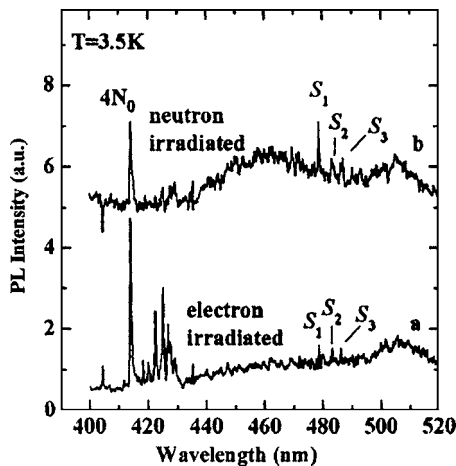


FIG. 2. LTPL spectra of *n*-type 6H-SiC after neutron- and electron-irradiation.

is a kind of secondary defect. However, no further information about their transition could be inferred since neither existed for quite a range of annealing temperature (500 °C–700 °C).

Although the  $D_1$  center in 6H-SiC was widely observed in the past, it was mostly in the context of ion implantation usually accompanied by high-temperature annealing (hence missing the low-temperature annealing features).<sup>2,13,16</sup> This may explain why only the  $D_1$  center was observed in their works.

Egilsson *et al.* reported a set of the PL spectra  $E_A$  in the region 4250–4500 Å in the electron-irradiated *n*-type 4H-SiC sample before annealing.<sup>17</sup> This set of defects which annealed out at approximately 750 °C may well be similar to the defects  $S_1$ ,  $S_2$ ,  $S_3$  reported here.

In previous works, the only defect with annealing temperature around 500 °C was the defect  $H_1$  detected using DLTS in electron-irradiated *p*-type 6H-SiC, which seemed to be similar to this observation of the  $S_1$ ,  $S_2$ ,  $S_3$ .<sup>18</sup> However, the  $H_1$  did not exhibit any character of inequivalent sites as does  $E_1$ ,  $E_2$ .

According to recent experimental and theoretical studies, the majority of initial defects introduced by irradiation were single vacancy ( $V_{Si}$  or  $V_C$ ) related.<sup>19–21</sup> In SiC, both the silicon and carbon vacancies are stable at room temperature.<sup>22</sup> Employing positron annihilation spectroscopy (PAS) measurement, Ling *et al.* pointed out that  $V_{Si}$  and  $V_C$  would disappear after a 450 °C to 600 °C annealing.<sup>23</sup> All these experimental results indicate that  $S_1$ ,  $S_2$ ,  $S_3$  are possibly generated from single vacancy defects ( $V_{Si}$  or  $V_C$ ), but their microstructures remain unidentified. Further studies on this topic are certainly necessary.

Many techniques, such as DLTS, PL, and PAS, have been employed to monitor the characteristics of irradiation-induced defects in SiC and a series of irradiation related defects have been reported.<sup>1–4</sup> Annealing behaviors of these defects were investigated in order to understand the relationships among results from different techniques. For example, the defects  $E_1$ ,  $E_2$  from DLTS and the  $D_1$  center were considered to be the same defect because of their similar thermal behaviors and character of inequivalent sites. However, as it has been pointed out, the family of irradiation-induced defects in 6H-SiC seem to be very complicated due to their different thermal behaviors.<sup>1,18</sup> The experimental results of this work have indicated that the  $D_1$  center was a secondary defect produced in the annealing process while the defect  $E_1$ ,  $E_2$  existed in the as-irradiated materials.

The PL lines at 419–430 nm are the emission lines of  $4N_0$  accompanied by its phonon replicas (as already reported).<sup>12,24</sup> As for the as-neutron-irradiated samples, we think that there may well be competing (recombination) defects shunting the PL signal in question. Upon annealing, many of these defects killing the PL signal get annealed out allowing the PL lines to appear. This can explain why these spectral features appear for the neutron-irradiated samples only after annealing. The broad feature between 440 nm and 480 nm is systematic noise. However, in 1993, Pensl and Choyke reported PL signals in this range,<sup>12</sup> the discrepancy was possibly due to the difference in SiC samples of the two

studies, since the SiC samples used by Pensl *et al.* were early SiC materials and had more defects induced by the growth of SiC crystals.

#### IV. CONCLUSION

In conclusion, a set of defects  $S_1$ ,  $S_2$ ,  $S_3$  has been observed in as-neutron (electron) -irradiated  $n$ -type 6H-SiC for the first time. These were completely annealed out after a 500 °C treatment. The  $D_1$  center did not appear until after 700 °C annealing. These experimental results strongly indicate that the  $D_1$  center is a secondary defect. Even with the very different thermal behaviors, it is still not possible to infer the microstructures of these defects and thus further studies are necessary.

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