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Formation and annihilation of intrinsic-related defect centers in high energy electron-irradiated or ion-implanted 4H– and 6H–silicon carbide

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Abstract

Intrinsic-related defect centers (IRDCs) in 4H–/6H–SiC are generated by implantation of helium ions, protons or by irradiation of high energy electrons. The formation and thermal stability of these centers are studied by deep level transient and positron annihilation spectroscopy subsequent to anneals at 600–1800°C. It turns out that the formation of IRDCs depends on the injected particle. Further we have identified defect centers which show identical temperature dependence of DLTS defect concentrations and positron capture rates. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: 4H–SiC; 6H–SiC; Intrinsic-related defect centers; DLTS; PAS

1. Introduction

Ion implantation, which is an important tool to dope silicon carbide (SiC) with donors and acceptors, not only causes lattice damage but is also accompanied by an unintentional local disturbance of the stoichiometry of SiC. Based on the different masses of silicon (Si) and carbon (C) atoms, the heavier Si atoms accumulate close to the surface while the lighter C atoms are knocked on deeper into the bulk. This effect may, in addition, enhance the formation of intrinsic-related defect centers (IRDCs). For a survey of IRDCs in ion-implanted or electron-irradiated SiC, see e.g. Refs. [1–3].

In this paper, we conduct a detailed study of the formation and annihilation of IRDCs in helium (He^+), proton (H^+) or 2 MeV electron (e^-) damaged and subsequently annealed n-type 4H–/6H–SiC. Deep level transient spectroscopy (DLTS) and positron annihilation spectroscopy (PAS) are applied to determine defect concentrations and positron capture rates, respectively. It is the aim to examine whether the formation of IRDCs differs for the different generation processes and whether part of the observed defects are vacancy-related.

2. Experimental

Nitrogen (N)-doped epitaxially grown SiC layers of 4H and 6H polytype, respectively, are used for these investigations (CREE, $[N_N - N_{Comp}] = 5 \times 10^{15}\,cm^{-3}$). Square-shaped samples with an area of 5 mm × 5 mm are cut from the wafers. Intrinsic defect centers are generated by irradiation of 2 MeV electrons (e^- current = 0.5 mA, fluence = $10^{15}\,cm^{-2}$) or by implantation of 2 MeV protons (Gaussian H^+-profile with $R_p = 25\,\mu m$

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(calculated by TRIM_C), H^+ current = 30 nA, fluence = 10^{13} cm^{-2}) or helium ions (box profile generated by multiple implantation of He^+ ions with energies ranging from 30 to 950 keV, He^+-current = 50 nA, total He^+ fluence = 8×10^{12} cm^{-2}). The e$^-$-irradiated and He^+-implanted samples are sequentially exposed to anneals at temperatures, T_a, ranging from 600°C to 1700°C for 30 min. They are characterized by DLTS subsequent to each heat treatment (similar processed samples were investigated by PAS, see Ref. [4]). In contrast each H^+-implanted sample is isochronally annealed for 30 min at one fixed temperature. Anneals up to 1000°C are performed under vacuum in a rapid isothermal annealing (RIA)-system. Above 1000°C the samples are put into a SiC container and annealed under 1 atm Ar pressure in a resistance-heated graphite furnace. Subsequent to heat treatments above 900°C, a surface layer of 100 nm is etched off by reactive ion etching (RIE). Schottky contacts are fabricated by evaporation of nickel (Ni) through a shadow mask (contact diameter varies between 0.35 and 1 mm); large area Ohmic contacts (Ni) are evaporated on the backside of the samples. Prior to each annealing step the contacts are removed. DLTS spectra are taken in a temperature range from 90 to 700 K. The filling pulse width is 20 ms and the rate window is chosen between 0.25 and 32 ms. Defect concentrations are determined from the height of the DLTS peaks.

3. Results and discussion

Although all the observed DLTS spectra show identical features (IRDC-related peaks), the abundance of defect centers and their formation strongly depend on the particular injected particles. Fig. 1 shows DLTS spectra taken on an e$^-$-irradiated and annealed ($T_a = 900°C$, $t_a = 30$ min) 4H– (solid curve) and 6H–SiC (dotted curve) epilayer, respectively. The dominating peaks in the spectrum of the 4H–SiC epilayer (related to the y-axis on the left) are attributed to electron traps like the ET1-, ET3- and Z_1/Z_2(4H)-center [5]. The activation energies and generated concentrations of these IRDCs are summarized in Table 1. Their chemical composition and microscopic structure are not known. Similar electron traps are described in Ref. [2]. In addition, the titanium (Ti) acceptor is observed at about 90 K. Ti is an omnipresent contamination in SiC due to graphite parts. In the case of the 6H–SiC polytype (related to the y-axis on the right), the E_1/E_2- and Z_1/Z_2(6H)-center prevail (for activation energies and concentrations, see Table 1).

The formation of the observed IRDCs in 4H–/6H–SiC strongly depends on the injected particles. As an example the annealing behavior of Z_1/Z_2(4H)-centers in n-type 4H–SiC generated by He^+-ions, H^+-ions and high energy electrons, respectively, is displayed in Fig. 2. The implantation/irradiation parameters are selected in such a way that (a) the He^+ implantation generates a vacancy profile to a depth of 2.2 μm with a mean

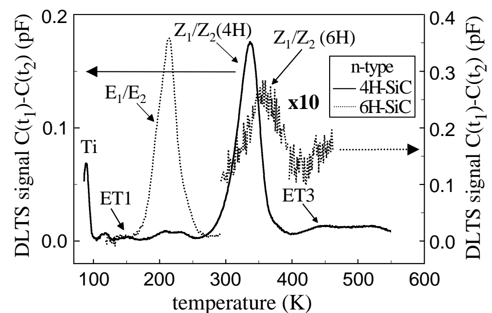

Fig. 1. DLTS spectra taken on an e$^-$-irradiated and annealed ($T_a = 900°C$, $t_a = 30$ min) 4H– (solid curve) and 6H–SiC (dotted curve) epilayer, respectively. The corresponding defect parameters are summarized in Table 1.

Table 1
Ionization energy, concentration and thermal stability of intrinsic-related defect centers generated by implantation of He^+-, H^+-ions and by irradiation of 2 MeV electrons in 4H–/6H–SiC

Defect	Ionization energy (meV) for $\sigma \sim T^0/T^{-2}$	Concentration (cm^{-3}) (subsequent to anneal at 900°C/30 min)	Thermal stability (°C)
4H–SiC			
ET1	220/245	6.1×10^{12}	RT–1300
ET3	660/740	1.9×10^{13}	RT–1300
Z_1/Z_2(4H)	590/650	2.3×10^{14}	400–1700
6H–SiC			
E_1/E_2	0.42/0.46	3.6×10^{13}	RT–1200 (?)
Z_1/Z_2(6H)	660/720	1.9×10^{13}	RT–1700

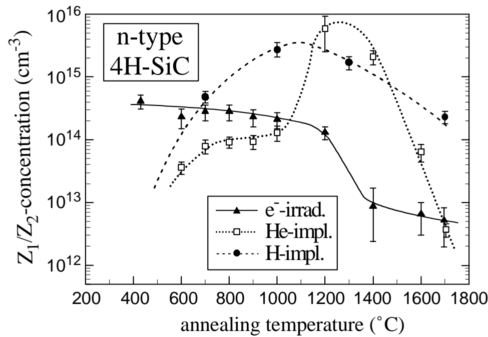

Fig. 2. Annealing behavior of Z_1/Z_2-centers in n-type 4H–SiC generated by implantation of He^+- and H^+-ions or by irradiation of 2 MeV electrons. The annealing time t_a at fixed temperature is 30 min. High Z_1/Z_2-concentrations are determined by temperature-dependent $C-V$ measurements.

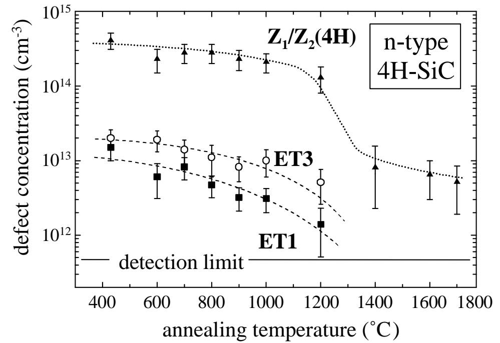

Fig. 3. Annealing behavior of ET1-, ET3- and Z_1/Z_2-centers in n-type 4H–SiC generated by irradiation of 2 MeV electrons. The annealing steps ($t_a = 30$ min at each applied temperature) have been sequentially conducted.

concentration of 2×10^{18} cm^{-3} (calculated by TRIM_C), (b) the H^+ ions accumulate in a depth of approx. 25 μm and can, therefore, not directly affect the formation of IRDCs in the investigated zone and (c) the 2 MeV electrons generate an almost homogeneous intrinsic defect concentration through the whole bulk of the SiC wafer. The temperature during ion implantation/e$^-$ irradiation is in each case below 50°C; this low temperature avoids annealing of lattice damage during irradiation. In He^+- and H^+-implanted samples, the Z_1/Z_2-center is not observable directly after the implantation; its formation requires an annealing step at temperatures above 600°C. The Z_1/Z_2-concentration increases with increasing annealing temperature and reaches a maximum value in the temperature range between 1100°C and 1200°C. This observation is also true for heavier ion masses (see Ref. [3]). Irradiation with 2 MeV electrons generates the Z_1/Z_2(4H)-center without additional anneal. We suggest that the number of displacements per injected particle is much lower for electrons than for ions. Therefore, isolated intrinsic defects must be formed in ion-implanted layers by additional heat treatments. Annihilation of the Z_1/Z_2(4H)-center occurs at temperatures $T_a > 1200$°C in all three cases.

Our experimental results are in contrast with data published by Storasta et al. [6], who reported that the maximum concentration of Z_1/Z_2(4H)-centers generated by H^+-ions (2.9 MeV) does not show any distinct change with the annealing temperature up to 1300°C.

The concentration of ET1, ET3 and Z_1/Z_2(4H)-centers as well as that of E_1/E_2- and Z_1/Z_2(6H)-centers in electron-irradiated n-type 4H– and 6H–SiC samples, respectively, as a function of the annealing temperature is displayed in Figs. 3 and 4. The annealing steps (30 min each) have been sequentially conducted. Up to 1000°C

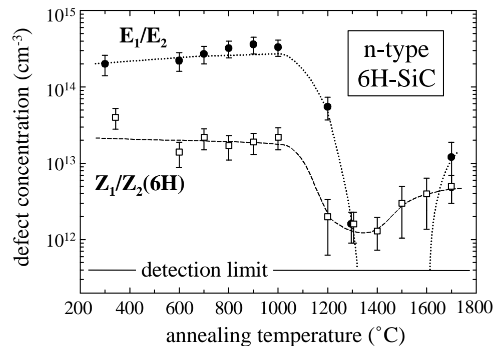

Fig. 4. Annealing behavior of E_1/E_2- and Z_1/Z_2-centers in n-type 6H–SiC generated by irradiation of 2 MeV electrons. The annealing steps ($t_a = 30$ min at each applied temperature) have been sequentially conducted.

the defect concentrations are not affected by the heat treatments. For temperatures $T_a > 1000$°C, the concentrations strongly decrease. For $T_a > 1300$°C, ET1- and ET3-centers are no longer detectable with our DLTS system. Z_1/Z_2(4H)-centers in the 4H polytype and E_1/E_2- and Z_1/Z_2(6H)-centers in the 6H polytype show qualitatively a similar annealing behavior. In the temperature range from 1300°C to 1600°C, the concentration of these centers is strongly reduced (the DLTS signal of E_1/E_2-centers is below the detection limit). Subsequent to anneals at $T_a > 1400$°C, these defect concentrations begin to increase again. They are still detectable at a few 10^{12} cm^{-3} subsequent to a heat treatment at 1700°C (for the temperature range of thermal stability of defects, see Table 1, last column). These results are reproducible and have been repeated with a series of samples. The formation and dissociation of intrinsic-related defects in this temperature range appear to be extremely complex. Most of the intrinsic-

related defect complexes are dissolved in this temperature range and serve as a source for Si and C interstitials and/or vacancies. This increase in intrinsic defects may lead to the observed slight recovery of Z_1/Z_2(4H), E_1/E_2- and Z_1/Z_2(6H)-centers.

PAS investigations on identically processed 6H–SiC samples (not shown here, see Ref. [4]) also result in a decrease of the positron capture rate of the particular defect centers at annealing temperatures above 1000°C. The clear temperature correlation between DLTS concentrations and positron capture rates strongly indicates that the centers under discussion (Z_1/Z_2(4H)-center in 4H–SiC, E_1/E_2- and Z_1/Z_2(6H) in 6H–SiC) are vacancy-related.

4. Summary

Intrinsic-related defect centers are generated in 4H–/6H–SiC by implantation of He^+-ions or protons or by irradiation of high energy electrons. The formation and annihilation of IRDCs is studied as a function of annealing temperature in the range from 600°C to 1700°C. The formation process of IRDCs depends on the injected particle. Implantation of ions (e.g. H^+, He^+) requires an annealing step above 600°C to form IRDCs. In the 4H– and 6H–SiC polytypes ET1, ET3, Z_1/Z_2(4H) and $E_1/E_2, Z_1/Z_2$(6H), respectively, are the dominating centers; they partially resist heat treatments up to 1700°C. In electron-irradiated samples, these defects are already present at maximum concentration without additional anneal. Based on the comparison of DLTS and PAS results taken on identically processed 4H–/6H–SiC samples, it is concluded that vacancies participate in the investigated defect complexes.

Acknowledgements

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