Journal of Electronic Materials, Vol. 21, No. 7, 1992

Electron Spin Resonance Study of Defects in CVD-Grown 3C-SiC Irradiated With 2MeV Protons

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Electron spin resonance (ESR) was used to study defects induced by 2MeV-proton irradiation in cubic silicon carbide (3C-SiC) epitaxially grown on Si substrates by chemical vapor deposition. A new ESR signal labeled T5 was observed at temperatures lower than ≈100 K in Al doped, *p*-type 3C-SiC epilayers irradiated. The T5 signal has anisotropic *g*-values of $g_1 = 2.0020 \pm 0.0001$, $g_2 = 2.0007 \pm 0.0001$, and $g_3 = 1.9951 \pm 0.0001$. The principal axes of the *g*-tensor were found to be along the ⟨100⟩ directions, indicating that the T5 center has D_2 symmetry. Isochronal annealing of the irradiated epilayers showed that the T5 center was annealed at temperatures around 150° C. A tentative model is discussed for the T5 center.

Key words: Electron spin resonance, silicon carbide, proton irradiation, defect, annealing

INTRODUCTION

Cubic silicon carbide (3C-SiC), which has zinc blende structure, is an excellent semiconductor for electronic devices used in high-temperature and radiation environments. Its desirable properties are ascribed to its large band gap, high electric breakdown field, high saturation drift velocity, moderately high electron mobility, temperature stability, and chemical inertness.[1] Single crystals of 3C-SiC were grown previously by the Lely process, but those obtained were of relatively small size and irregular shapes. This impeded modern electronic application of 3C-SiC. Recent technical advance in heteroepitaxial growth of 3C-SiC on Si substrates by way of chemical vapor deposition (CVD) led to production of single crystalline 3C-SiC films with large areas enough to their application to planar-structure devices.[2–4] This stimulated practical studies concerning 3C-SiC device fabrication.[5,6]

For the application of 3C-SiC epilayers to devices operated in a radiation field, it is important to clarify behavior of defects induced by a radiation. The knowledge of these defects is also needed for an understanding of defects introduced by ion implantation in 3C-SiC. In recent years, data on radiation induced defects in 3C-SiC epilayers have been accumulated by photoluminescence,[7] deep-level transient spectroscopy,[8] nuclear reaction and backscattering,[9] and electron spin resonance (ESR)[10,11] measurements. However, only a little has been revealed about structures and electronic levels of radiation induced defects in 3C-SiC up to now.

In this paper, ESR studies have been performed of the defects introduced by 2MeV-proton irradiation in *p*-type 3C-SiC epilayers grown by a CVD method. We show a new ESR signal which consists of three ESR lines and additional weak ESR lines. A tentative model of the defect is discussed based on the analysis of the Zeeman and hyperfine (hf) interactions obtained from ESR spectra. Isochronal annealing of this ESR center is also described.

EXPERIMENTAL

Single crystalline 3C-SiC layers with thicknesses of ≈30 μm were grown epitaxially at 1400° C on Si (100) substrates by means of CVD using SiH_4-C_3H_8-H_2 gas system. The details of the growth procedure have been described elsewhere.[3] Since as-grown, non-doped epilayers showed *n*-type electrical conduction, Al impurities were doped in 3C-SiC by a mixture of $(CH_3)_3Al$ in the growth gases so as to grow *p*-type samples. Hole densities in *p*-type 3C-SiC grown in this way were about $1 \times 10^{17}/cm^3$ at room temperature (RT). After the growth, the Si substrates were etched off with an HF-HNO_3 solution. The 3C-SiC epilayers were irradiated with 2MeV-protons up to the fluence of $1 \times 10^{16} H^+/cm^2$, where the proton flux was about $3 \times 10^{12} H^+/cm^2 sec$ (≈$0.4 \mu A/cm^2$). The samples were placed on a heat sink so that their temperatures kept below 50° C during the irradiation.

ESR measurements of the irradiated samples were performed over a temperature range from 4K to RT with an *X*-band microwave incident upon TE_{011} cylindrical cavity. 3C-SiC samples were rotated around the ⟨011⟩ axis perpendicular to the magnetic field in order to obtain the angular dependence of ESR spectra. Isochronal annealing of the epilayers irradiated was also made for 5 min in flowing pure Ar gas.

(Received August 8, 1991; revised February 11, 1992)

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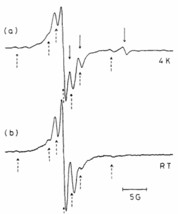

Fig. 1 — ESR spectra (a) at 4K and (b) at room temperature of p-type 3C-SiC irradiated with 2MeV-protons of $1 \times 10^{16}H^+/cm^2$. Magnetic field was applied parallel to the ⟨100⟩ axis. The T5 and T1 centers are indicated by the solid-line arrows and the broken-line arrows, respectively.

RESULTS AND DISCUSSION

Figure 1(a) shows a typical ESR spectrum at 4K of the p-type 3C-SiC epilayers irradiated with protons of $1 \times 10^{16}H^+/cm^2$, when the magnetic field was applied parallel to the ⟨100⟩ axis ($H//\langle 100\rangle$). Several ESR lines were observed in the irradiated samples as indicated by the arrows whereas those were not seen in as-grown samples. Some of them were not observed at temperatures above ≈100K and the others remained even at RT, which are shown in Fig. 1(b). The dependence of the ESR line intensity on the microwave power also indicated a difference in the saturation behavior between the ESR lines represented by the solid-line arrows and those by the broken-line arrows in Fig. 1(a): The ESR lines indicated by the broken-line arrows were saturated and broadened at lower microwave powers than those by the solid-line arrows did. These results show that two kinds of ESR centers were induced by the irradiation.

The ESR signal observable at RT consists of central five ESR lines equally spaced at about 1.5G and satellite lines symmetrically disposed on both sides of the central lines. The position of the most intense central line indicated an isotropic g-value of 2.0028 ± 0.0002. Since the g-value, the separation width, and the angular dependence of these ESR lines were almost the same as those obtained for the T1 signal reported in a previous paper,[11] this signal is considered to be identical with the T1. The T1 signal, which was first observed in n-type epilayers irradiated with

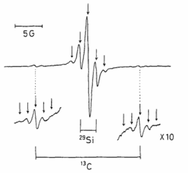

Fig. 2 — ESR spectrum at 10K of n-type 3C-SiC epilayers irradiated with $1 \times 10^{16}H^+/cm^2$, when the magnetic field was applied parallel to the ⟨100⟩ axis. The arrows indicate T1 signal whose satellite lines are due to the hyperfine interactions with ^{13}C and ^{29}Si nuclei.

protons as shown in Fig. 2, is interpreted by simultaneous hf interactions of a paramagnetic electron (or electrons) with ^{13}C (nuclear spin $I_C = 1/2$, natural abundance 1.1%) at four nearest-neighbor carbon sites and ^{29}Si (nuclear spin $I_{Si} = 1/2$, natural abundance 4.7%) at twelve second-nearest-neighbor silicon sites. It was also deduced from the angular dependence of the T1 signal that the hf interaction with ^{13}C is axially symmetric around the ⟨111⟩ axis. These give evidence that the T1 signal arises from an isolated vacancy at a silicon site.[11]

The ESR signal observed only below ≈100K consists of three ESR lines mainly in the case of H//⟨100⟩, as indicated by the solid-line arrows in Fig. 1(a). This ESR signal newly observed in p-type 3C-SiC is referred to as T5. No T5 signal was detected significantly in the proton irradiated n-type epilayers with the initial electron densities of $\approx 1 \times 10^{16}/cm^3$ at RT. Figures 3(a), (b), and (c) show the ESR spectra at 10K of the p-type epilayers irradiated with $1 \times 10^{15}H^+/cm^2$ when the magnetic field was applied parallel to the ⟨100⟩, ⟨111⟩, and ⟨011⟩ axes, respectively. In these spectra, broadening of the T1 signal took place but that of the T5 signal did not so that the ESR line intensity of the T5 signal is emphasized. It should be noted that the weak ESR lines were disposed on either side of the main lines, as indicated by the broken-line arrows in Fig. 3. The positions of the main three lines and the satellite lines were dependent on the direction of the magnetic field.

The intensity ratio of all the satellite lines to the main lines was 0.185 ± 0.02, which is about four times as large as the ^{29}Si abundance (4.7%). This suggests that the satellite lines are caused by the hf interaction of a paramagnetic electron with a ^{29}Si at four silicon sites, indicating a fourfold coordina-