Effect of heavy proton and neutron irradiations on epitaxial 4H-SiC Schottky diodes

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Abstract

In this work we report electrical characterizations on heavily irradiated epitaxial 4H-SiC Schottky diodes. Even after an irradiation at a fluence of $1.4 \times 10^{16}$ p/cm\textsuperscript{2} and $7 \times 10^{15}$ n/cm\textsuperscript{2}, we found the diodes still able to detect $\alpha$ and $\beta$ particles with a charge collection efficiency (CCE) ranging from 25 to 30% after proton irradiation and about 18% after neutron irradiation, at the highest reverse bias applied. This corresponds to a charge collection distance (CCD) of 7\,\mu m after the proton irradiation and 5\,\mu m after the neutron irradiation. As the irradiation level approaches the range $\sim 10^{15}$/cm\textsuperscript{2}, the material behaves as intrinsic due to a very high compensation effect.

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1. Introduction

Silicon carbide (4H-SiC) radiation detectors are being developed for a variety of applications including particle detection [1,2], radiation dosimetry [3], X-ray [4] and UV detection [5]. The wide bandgap of 4H-SiC (3.26\,eV) makes it an attractive semiconductor, capable of stable operation in...
elevated temperature and intense radiation environments [6]. Radiation hardness studies on SiC sensors have been performed recently by means of irradiations of neutrons [7], electrons [8], protons and gamma rays [9,6], pions [10] and light ions [11]. It has been suggested [10] that operation of SiC detectors at elevated temperatures will prolong SiC detector operating lifetime. The influence of thermal annealing on radiation-induced defects has been studied recently [12,13]. A strong radiation-induced compensation has been observed after fluences of $10^{15}$ cm$^{-2}$ (8.6 MeV) electrons. Subsequent thermal annealing stages up to 470 K are effective in increasing the free carriers concentration of the SiC detectors, but do not change their charge collection performances [14].

In this work we report, for the first time to our knowledge, results of the radiation-induced degradation in performance of epitaxial Schottky diodes at proton and neutron fluences comparable to that undergone by vertex detectors, over their projected lifetime in future high energy physics experiments at CERN [15]. The samples were investigated by means of $C-V$ measurements, $\alpha$ and $\beta$ charge collection measurements, before and after irradiation. An investigation of radiation-induced defects on the same batch of samples is underway.

2. Devices and experimental procedure

2.1. Detectors

Tested devices are Schottky diodes manufactured by Alenia Marconi Systems (Rome) on epitaxial (4H-)SiC grown by IKZ (Berlin) and CREE Research (USA). The high quality epitaxial layer with a thickness ranging from 20 to 50 $\mu$m was grown by Chemical Vapour Deposition (CVD) on a more defective substrate manufactured by CREE Research. The substrate is nitrogen-doped n$^+$ type and the epitaxial layers are n-doped at different net nitrogen densities (see Table 1). The epitaxial layer thicknesses were measured, before irradiation, by optical interferometry (with a procedure described in Ref. [1]) and are listed in Table 1. A former batch of detectors was manufactured using gold contacts for the Schottky barrier [8], whilst a Ni$_2$Si Schottky contact was coated on the most recent material [1]. In both cases an ohmic contact was obtained by the deposition of a multilayer of Ti/Pt/Au onto the backside of the substrate. Thick (5 $\mu$m) gold circular dots with a diameter of 60 $\mu$m were grown onto the diode dot and the guard ring for their electrical (wire bonding) connection to the instruments. Those can be seen in Fig. 1, showing a sample with three Schottky detectors. The bigger contacts were mainly used in this work (2.52 mm diameter).

2.2. Experimental setup

2.2.1. Capacitance vs. voltage setup

$C-V$ measurements have been performed in a white chamber (class 10,000), using a probe station interfaced with an HP 4284A LCR meter and an HP 4142B generator, providing capacitance measurements in the range 20Hz–1 MHz. We were
limited in the range to 100 Hz–1 MHz by the signal to noise ratio. The detector performances have been tested by 5.486 MeV α-particles from a \(^{241}\)Am source in vacuum (about 1 Pa) and in air by MIPs from a \(^{90}\)Sr source.

2.2.2. Charge collection measurement setup

The response of the sample from electrons from a \(^{90}\)Sr source was measured with a characterization setup using a low noise preamplifier having a 2.4 μs shaping time [16]. The particles from the \(^{90}\)Sr source are confined by a plastic collimator to a beam of about 1 mm wide. After having passed the sample they hit a small scintillator that is coupled to a photomultiplier. The dark count rate of the scintillator setup is of the order of 2 hits per minute, while the trigger rate during data taking was of the order of 20–30 Hz. Still we normally observe pedestal events of the order of 4%, also when the sample is much wider than the particle beam. Therefore, we assume that the additional pedestal events originate from bremsstrahlung interactions in the sample. These are strongly dependent on the source–surface distance, most likely because of stray X-rays originated by the β radiation. Hence the pedestal fraction can vary in different measurements or with different sample thicknesses.

For each measurement at least 10,000 events were collected and histogrammed. The bin width of the histograms corresponded to a charge of 220 electrons or 110 for narrow histograms. Through the histogram an empirical Landau curve was fitted [17] that has been convoluted with a Gaussian function representing the electronic noise of ENC = 280 + 10/e (measured with calibrated capacitors for the present work). A Gaussian pedestal peak was added to the convoluted Landau curve, with a linewidth of about 350 electrons that was centered on the pedestal value. The Gaussian width was measured independently at each bias value, by acquiring false events, triggering the acquisition system with a pulse generator. The resulting curve accounted for the zero-signal events originating from bremsstrahlung.

Subsequently, for a few measurements with the highest signal level, which is as low as 600 electrons for the irradiated samples, the fraction of pedestal events was obtained, by fitting the data with the sum of the pedestal peak and the Landau. For some applied bias values, it was necessary to retune the pedestal value a bit. Accordingly, values between 2.5 and 6% were found, for the fitted fraction of pedestal events, which is well in agreement with earlier characterizations on samples with similar thickness and geometry. The averaged value of the fitted Landau curve was used as the outcome of the analysis. For measurements of a lower charge signal the pedestal peak became completely buried in the Landau curve so the analysis was done with an assumed fraction of 4% pedestal events.

The guard ring has not been connected during the charge collection measurements.

The detector performances have also been tested by 5.486 MeV α-particles from a \(^{241}\)Am source in vacuum (about 1 Pa), with another charge collection setup. The range of the alpha’s in SiC is 18 μm. Since the incident radiation is stopped inside the sample, we can no longer use the trigger system previously described. The trigger is given by the signal itself at a fixed threshold level.

Details on the two charge collection characterization systems can be found in Refs. [18,19].
Note that in $z$ measurements the carriers are generated within the range and one type of carrier can drift toward the source an average distance limited by the range itself, while the other carrier can drift from the generation point to the opposite electrode, i.e., the upper limit is the active thickness of the sample. Instead, with $\beta$-particles, the situation is symmetric for both carriers, since a uniform generation rate is assumed through all the sample. In both cases the Charge Collection Distance (CCD) is defined as the distance the carriers drift apart before being trapped or reaching the electrodes, which is often quite different from the sum of the mean free paths of electrons and holes. Defining the Charge Collection Efficiency (CCE)

$$CCE = \frac{Q}{Q_0}$$

where $Q$ is the charge induced at the electrodes and $Q_0$ is the charge generated by the radiation probe, from Ramo’s theorem we have that

$$CCD = CCE \times (\text{active thickness})$$

### 2.2.3. Sensitivity of the charge collection setup

Fig. 2 shows the electronic pedestal of sample R0501-04 # 21 (CREE), which is the about same at all bias values, since as will be seen later, the capacitance of the sample is independent of voltage. The fit to a Gaussian curve gives a width of 1.78 mV which results in an ENC $\approx 400$ e$^-$. This value is in agreement with a capacitance of the order of 10 pF, which is correct for this batch of samples (see Section 3.2.1). We can use this noise value to fit the pulse height spectra. Fig. 3 shows the spectrum collection of the same sample at a bias of 600 V. The fit is quite good using the noise value determined. The mean value of the deconvoluted Landau is about 650 e$^-$ and the fraction of pedestal events is found to be 2.5%. The pedestal and the signal are not separated even at high voltages. At lower voltages the situation is depicted in Fig. 4 showing a measurement at $-100$ V. A pedestal fraction of 4% was used in the fit. The mean value obtained is about 280 e$^-$. This value can be only indicative. We cannot extract the signal. However, we observe a definite trend: the charge collected increases with increasing applied field.

![Fig. 2. Fit of the electronic noise to a Gaussian lineshape for sample CREE # 21. The width is about 1.8 mV which results in an ENC $\approx 400$ e$^-$. This noise value has been used for the fit of the pulse height spectra.](image2)

![Fig. 3. Pulse height spectrum for sample CREE # 21 at 600 V. The pedestal fraction used in the fit is 2.5%. The deconvoluted Landau is drawn with a dotted line. The fit and the pedestal are drawn with a solid line. The mean value of the deconvoluted Landau is about 650 e$^-$.](image3)

### 2.3. Irradiation

Sample named SiC038105 # G1 has been irradiated with a fluence of $1.4 \times 10^{16}$/cm$^2$ 24 GeV protons at the CERN Proton Syncrotron (PS). Samples from the batches N31-39 (IKZ) and
R0501-04 (CREE) have been irradiated with increasing fluence of 1 MeV (NIEL equivalent in Si) neutrons at Neutron Irradiation Facility in Ljubljana of the Jozef Stefan Institute. Details about the nuclear reactor used (TRIGA), the irradiation setup and the neutron spectrum can be found in Ref. [20]. The neutron irradiations levels used range from $3 \times 10^{13}$ to $7 \times 10^{15}$ cm$^{-2}$.

### 3. Experimental results

#### 3.1. Characterization before irradiation

Samples of the batch SiC038105 have been fully characterized before irradiations, and the results have been presented in a previous work [21]. The response of the SiC devices yields a depletion width of about 20 $\mu$m by $C$–$V$ measurements. They are characterized by a 100% CCE, by $x$ and $\beta$-MIP charge collection measurements: The charge signal is stable and reproducible, with no evidence of priming or polarization effects.

Samples from the more recent batches N31-39 (IKZ) and R0501-04 (CREE) have been characterized in the present work. Fig. 5 shows the $C$–$V$ curve of sample IKZ # 08 taken at 10 kHz. It was not possible to fully deplete the diode, as breakdown occurs at about 550 V. The linear fit to the curve yields a (mean) value of net nitrogen doping of $N_{\text{eff}} = 5.0 \times 10^{14}$/cm$^3$ (see also Table 1). The corresponding $\beta$ charge collection measurements yield a collected electron equivalent charge of $(1400 \pm 200)$ e$^{-}$ at 200 V. The CCD is given for the $\beta$ MIPs by the collected charge number divided by the number $N$ of generated pairs per $\mu$m. Assuming a value of $N = 55$ pairs/$\mu$m for 4H-SiC [1], the CCD of the carriers is $(25 \pm 4)$ $\mu$m. From the $N_{\text{eff}}$ value we can easily calculate a depletion width of 21 $\mu$m at the abovementioned voltage. Thus, we can conclude that we have a 100% CCE from 25 $\mu$m and 23 $\mu$m, respectively. At higher voltage the leakage current was too high (more than 5 nA) and the signal to noise ratio too low to perform reliable measurements. That limit is probably due to surface effects, since in measurements performed in a slight overpressure of nitrogen (used to avoid moisture) we observe usually leakage currents an order of magnitude (or more) smaller than in air. The net doping level of the R0501-04 (CREE) batch, before irradiation, was found in the range $3.6-4.4 \times 10^{14}$/cm$^3$ by $C$–$V$ analysis (Table 1). This implies a depletion width of $(16 \pm 1)$ $\mu$m at 100 V reverse bias and $(23 \pm 1)$ $\mu$m at 200 V. Two samples of the batch were measured with the $\beta$ CCD setup: CREE # 88 and CREE # 86. The CCD measured at 100 V (the
The highest bias value for sample CREE # 88 is $(18 \pm 2) \mu m$ and that at 200 V (the highest bias value) for sample CREE # 86 is $(26 \pm 2) \mu m$. Again we conclude that we observe 100% CCE.

3.2. Characterization after irradiation

3.2.1. C–V results

Fig. 6 shows the C–V curve of sample (IKZ # 32) after the $3 \times 10^{15}/cm^2$ neutron irradiation of Table 1. The capacitance is constant with voltage. The value is $8.45 \mu F$, and the fluctuations observed in Fig. 6 are not significant. We found the same result at different frequency from 100 Hz to 10 MHz. The CREE sample # 32, irradiated at a fluence of $7 \times 10^{15}/cm^2$, also yields a constant capacitance of $C = 10.9 \mu F$. Considering the dot diameter value ($2.52 \mu m$), these values of capacitance result in thicknesses of $50 \mu m$ (IKZ # 32) and $39 \mu m$ (CREE # 32), i.e., very close to the value obtained for non-irradiated samples (see Table 1). The same is true for the proton-irradiated sample (CREE # G1). With a contact diameter value of $2.0 \mu m$, a value of $C = 10.8 \mu F$ yields a thickness of $24.5 \mu m$. Hence, after the proton irradiation or the (lower fluence) neutron irradiation, the samples have become intrinsic, with a capacitance equal to the geometrical value, evaluated with the whole epitaxial layer thickness.

3.2.2. $\alpha$ charge collection results

The charge collection measurements with $\alpha$ particles have been performed over a wider range of irradiations than that presented in Table 1. Fig. 7 shows the CCD vs. bias voltage, at different neutron irradiations, from $2 \times 10^{13} \text{n/cm}^2$ to $7 \times 10^{15} \text{n/cm}^2$. We observe a strong decrease in the charge collection efficiency, at a fluence level of $\sim 10^{15}/cm^2$.

3.2.3. $\beta$ charge collection results

Fig. 8 shows the CCD of sample CREE # G1 obtained by $\beta$ MIP charge collection measurements. The collection efficiency is reduced from $25 \mu m$ to about $30\%$ at the highest voltage. From the $\beta$ measurements, the charge collection seems degraded of the same extent in irradiation by neutrons and by protons (see Fig. 11). This can also be observed in Fig. 9, which shows the CCD curve of sample CREE # 21, irradiated at a fluence of $3 \times 10^{15} \text{n/cm}^2$. The CCD is calculated from the electron charge collected $Q$ and the number of induced e–h pairs per MIP per $\mu m$, $\mathcal{N} = 55 [1]$: \[ \text{CCD} = \frac{Q}{\mathcal{N}}. \]

The material has become intrinsic and the response is of the same order for both bias polarities. Fig. 10 shows the CCD curve of sample CREE # 32 irradiated at the highest fluence ($7 \times 10^{15} \text{n/cm}^2$). The response has dramatically decreased. At the highest voltage (600 V), samples IKZ # 32 ($3 \times 10^{15} \text{n/cm}^2$) and IKZ # 55 ($7 \times 10^{15} \text{n/cm}^2$) have become intrinsic.
10^{15} \text{n/cm}^2 \) exhibit values as low as 650 e\textsuperscript{−} and 300 e\textsuperscript{−}, respectively. In Fig. 11 we have reported for comparison the \( \alpha \) and \( \beta \) measurements of CREE samples, after neutron irradiations.

4. Discussion and conclusion

The main findings in the present work are the following:

- Even after an irradiation at a fluence of \( 1.4 \times 10^{16} \text{p/cm}^2 \) and \( 7 \times 10^{15} \text{n/cm}^2 \), the diodes were still able to detect \( \alpha \) and \( \beta \) particles with a CCE ranging from 25\% to 30\%, corresponding to a charge collection distance of 7\mum for proton irradiation, and about 18\%, corresponding to a CCD of about 5\mum for neutron irradiation, at the highest reverse bias supplied. Similar results are found by Ruddy et al. [7] on 4H-SiC pn diodes after extreme neutron irradiation.

- However, the response after irradiation is quite low, considering that the epitaxial layer is thin. Nonetheless, it is clear that the performances are dependent on the material and on the preparation of the device, i.e., the CREE ARTICLE IN PRESS

Fig. 8. \( \beta \) particle CCD curve of sample CREE \# G1 irradiated at a fluence of \( 1.4 \times 10^{16} \text{p/cm}^2 \).

Fig. 9. \( \beta \) CCD curve of sample CREE \# 21 irradiated at a fluence of \( 3 \times 10^{15} \text{n/cm}^2 \). The material has become intrinsic and the response is of the same order for both bias polarities.

Fig. 10. \( \beta \) CCD curve of sample CREE \# 32 irradiated at a fluence of \( 7 \times 10^{15} \text{n/cm}^2 \). The response has dramatically decreased.

Fig. 11. \( \beta \) and \( \alpha \) CCD curves of samples CREE irradiated at fluences of 3 and \( 7 \times 10^{15} \text{n/cm}^2 \) and \( 1.4 \times 10^{16} \text{p/cm}^2 \).
samples yield a better response in terms of leakage current and CCD than the IKZ ones. Moreover, the quality of the material and of the devices is continuously increasing and new measurements are needed to validate this preliminary work.

- We have observed a change of the material to intrinsic, with a symmetric CCE response at both polarities and a flat $C-V$ response, as the irradiation level approaches the range $\sim 10^{15}$/cm$^2$. Actually the material is behaving as intrinsic because it is highly compensated. Compensation of 4H-SiC Schottky devices due to radiation damage has already been reported [12]. A further study is underway to investigate the nature of the radiation-induced defects responsible for this strong effect.

References