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### Silicon carbide PIN diode detectors used in harsh neutron irradiation



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#### ABSTRACT

Silicon carbide (SiC) neutron detectors based on PIN diode with Ti/Au electrode structure have been successfully developed and considered to be a good option for neutron detection related to harsh radiation environment, but these detectors suffered inevitable irradiation damage and performance degradation, ultimately damaged the applications severely. Unfortunately, the systematic research focusing on the degradation and irradiation defects is lack, and the mechanism of degradation is not clear yet. In this paper, these issues are carefully studied: SiC detectors were irradiated by neutrons emitted from fusion neutron generator and nuclear reactor with fluence ranging from  $1 \times 10^{14}$  n/cm<sup>2</sup> to  $2 \times 10^{16}$  n/cm<sup>2</sup>, then the performance degradation and neutron induced defects were carefully investigated. It is concluded that the SiC detectors could endure neutron irradiation as least 10<sup>16</sup> n/cm<sup>2</sup>. Although neutron irradiation could induce breakdown of PN junction, decrease of charge collection efficiency and worsening of energy resolution, all the detectors are still alive; furthermore, they could still acquire integrated response spectra to alpha particles with neglectable increase of dark current at neutron fluence of  $2 \times 10^{16}$  n/cm<sup>2</sup>. The di-carbon antisite defects and vacancy-type defects induced by neutron irradiation should be responsible for the performance degradation, according to low temperature photoluminescence and positron annihilation Doppler broadening measurements. Possible degradation mechanism was discussed as well. © 2018 Elsevier B.V. All rights reserved.

#### 1. Introduction

Since the first Silicon Carbide (SiC) neutron detector for nuclear reactor monitoring was developed 60 years ago [1–3], the advantages of SiC detector have been long recognized, such as the capability to operate at elevated temperatures [4–6], better radiation resistance [7] than conventional Silicon or Germanium detectors and excellent neutron-gamma discrimination [8,9] origins from thin active regions. With the demonstration of technologies for producing high-quality SiC, both chemical vapor deposition (CVD) and large-wafer form in 1990'1990's, high quality CVD epitaxial SiC detectors were first successfully fabricated by F. Ruddy [10] and Nava [11], obtaining good radiation perfor-

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https://doi.org/10.1016/j.sna.2018.07.053 0924-4247/© 2018 Elsevier B.V. All rights reserved. mance in energy resolution and charge collection efficiency (CCE), but in tiny dimension. After long-term work, high quality large area SiC detectors have been successfully fabricated by our group, with sensitive area reaching up to 4 cm<sup>2</sup>, analogous dimension to commercial silicon detectors [12]. Further challenge would focus on increasing the sensitive thickness of high quality SiC detector, giving it more potential in fast neutron detection (e.g. D–T fusion neutron detection using polyethylene converter) and high energy charged particle detection.

Neutrons, being electrically neutral, can usually be detected by using a converter foil containing  ${}^{6}Li$ ,  ${}^{10}B$ ,  ${}^{235}U$ , etc. The neutroninduced nuclear reactions lead to detectable ionization within the detector active volume, then multiple electron-hole pairs are generated in the depletion region of the SiC detector via coulomb scattering, and response signal is ultimately acquired by collection of electrons and holes. The ionization derived from reactions between neutrons and converter( ${}^{6}Li$ ,  ${}^{10}B$ ,  ${}^{235}U$ ) are  ${}^{3}H$  (2.73 MeV) +  ${}^{4}He$  (2.05 MeV),  ${}^{7}Li$  (0.84 MeV) +  ${}^{4}He$  (1.47 MeV), and low-mass fragment (95 amu, 93.0 MeV in average) + heavy-mass fragment (139 amu, 56.6 MeV in average), respectively, which have the max

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projected range in 4H-SiC material lower than  $30 \,\mu$ m. Fortunately, SiC Schottky diodes and PIN diodes with thickness ranging from 20 to  $100 \,\mu$ m have been successfully developed in recent years, fully meeting the requirement of neutron detection. By using 4H-SiC based diode detector, D Szalkai et al successfully detected 14 MeV neutrons in high temperature up to  $500 \circ [13]$  and B. Zat'ko et al detected thermal neutrons [14]. SiC detectors have been used in neutron flux monitoring [15–17] and proven to be with good radiation resistance [18,19].

SiC is considered to be with good radiation resistance, the threshold energy for defect production in SiC is considered to be larger than Si and GaAs by a factor of 1.5-2, of course is smaller than diamond by a factor of 2–2.5 [21]. But it is worth noting that the SiC neutron detectors suffered inevitable irradiation damage and performance degradation, badly influence their performance in the applications. As a result, investigation focusing on the performance degradation in neutron radiation is very important for SiC neutron detectors, seeking for suitable applications and evaluating the measurement accuracy. Most research of neutron radiation effects on SiC detectors is focused on Schottky diode detectors, getting neglectable signal deviation at a fluence of 10<sup>12</sup>-10<sup>13</sup> n/cm<sup>2</sup> [22,23], degradation of Schottky contact at a fluence of 10<sup>14</sup> n/cm<sup>2</sup> [24,25] and significant decrease of CCE at a fluence of 10<sup>16</sup> n/cm<sup>2</sup> [20,26]. However, the study of radiation effects on PIN type SiC detectors is insufficient. J Park et al. compared I-V characteristics, alpha energy spectra and signal current of PIN-type SiC detectors (29.7 µm in thickness) with Ti/Au and Cr/Au electrode structure with fluence ranging from 10<sup>15</sup> to 10<sup>17</sup> n/cm<sup>2</sup>, finding the Ti/Au structure devices with excellent radiation tolerance [27]. F. Moscatelli et al. irradiated a PIN diode (5 µm in thickness) with 1 MeV neutron with fluence in the range of  $10^{14}$ – $10^{16}$  n/cm<sup>2</sup> obtaining a decrease of leakage current density and doping concentration, and getting a very low recovery of radiation damage after anneal at 200° [28,29]. This work has successfully given a proof of high radiation resistance of SiC PIN-type neutron detector, but these research work is lack of systematic, the limited data is far from giving suitable applications reference. Furthermore, some important characteristics (e.g. CCE) have not even been discussed and origins of the degradation has not been identified.

In the present study, radiation resistance of SiC PIN-type detector (thickness:  $20-30 \,\mu$ m), which is irradiated by D–T fusion neutrons with fluence of  $1-7 \times 10^{14} \,n/cm^2$  and neutrons from Xi'an pulsed reactor with fluence of  $7-200 \times 10^{14} \,n/cm^2$ , has been investigated. Then the performance degradation was evaluated, such as forward I–V, reverse I–V, C–V, alpha particle spectra and CCE. Defects induced by neutron irradiation were characterized and the possible degradation mechanism was discussed as well.

#### 2. Experimental

#### 2.1. Sample preparation

PIN type 4H-SiC detectors mentioned here were fabricated with high-quality lightly doped epitaxial 4H-SiC layers (thickness:  $20-30 \,\mu$ m, target nitrogen doping concentration lower than  $1 \times 10^{14} \, \mathrm{cm}^{-3}$ , prepared by Nanjing Electronic Devices Institute, China) grown by chemical vapor deposition on commercial 4H-SiC N+ conducting substrate wafers ( $\Phi$  4 in. × 350  $\mu$ m, target nitrogen doping concentration of  $10^{19} \, \mathrm{cm}^{-3}$ , supplied by TankeBlue Semiconductor Co. Ltd., China). The front electrodes were fabricated after aluminum implantation and made of Ni/Au ( $100 \, \mathrm{nm}/2 \, \mu$ m). The aluminum ions were injected into SiC epitaxial layer with energy no more than 200 keV and then annealled at 1650°, getting a doping concentration of  $3 \times 10^{18} \, \mathrm{cm}^{-3}$ . The back ohmic contact electrodes were of ohmic contact, made with Ni/Au ( $100 \, \mathrm{nm}/3 \, \mu$ m)



**Fig. 1.** Schematic diagram of a PIN type 4H-SiC detector, which includes four layers: the Ni/Au layer in orange is the ohmic back electrode, the N+substrate in blue is the commercial 4H-SiC N+conducting substrate wafer, the N- layer in yellow is the sensitive volume of the SiC detector composited by high-quality lightly doped epitaxial 4H-SiC material, and the Ni/Au layer in green is the front electrode made on Al implantation layer in purple (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article).

annealed at 900°. Fig. 1 shows the schematic diagram of the PIN type 4H-SiC detectors.

Material-I and Material- II are lightly doped epitaxial 4H-SiC material ( $20-30 \mu m$ ) with commercial 4H-SiC N + conducting substrate ( $350 \mu m$ ), with a dimension of  $8 mm \times 8 mm$ . These two samples were prepared by Nanjing Electronic Devices Institute (China).

#### 2.2. Neutron irradiation

The neutron irradiation was performed at two neutron sources: the K600 Neutron Generator in China Institute Atomic Energy (CIAE) in Beijing (China), providing a constant deuterium-tritium fusion beam with an average energy of 14 MeV and a neutron fluence rate of  $4-12 \times 10^9$  n/cm<sup>2</sup>s; Xi'an pulsed reactor in Northwest Institute of Nuclear Technology (NINT) in Xi'an (China), providing 31% thermal neutrons and 69% fast neutrons. Five detector samples and a piece of material were irradiated by neutron supplied by these two neutron sources at room temperature, with experimental details shown in Table 1.

#### 2.3. Electric parameter measurement

The front I–V and C–V curves (acquired at 1 MHz) were measured with IWATSU CS-3200C Curve Tracer (Sample-25#, 23#, 22#, 24#) and Keithley 4200 Semiconductor Characterization System (Sample-6#, R201601, R201603).

The reverse I–V curves were measured in a copper box by Keithley-6517A pico-ampere meter at a reverse bias voltage ranging from 0 to 600 V, supplied by PS350 high voltage supply (Stanford Research System, Inc.).

#### 2.4. Alpha particle detection

The SiC detectors were used to detect alpha particles emitted by  $^{239}$ Pu alpha source ( $E_{\alpha}$  = 5.157 MeV [73.3%], 5.144 MeV [16.1%], 5.105 MeV [11.5%], 2  $\times$  10<sup>4</sup> Bq and 4  $\times$  10<sup>5</sup> Bq) in a vacuum chamber. The response signal induced in SiC detector was in turn transmitted to the following equipment through coaxial cables: an Ortec 142B preamplifier or ENDicott Interconnect EV550 preamplifier, an

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