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# Highly sensitive x-ray detectors in the low-energy range on n-type 4H-SiC epitaxial layers

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Schottky diodes on n-type 4H-SiC epitaxial layers have been fabricated for low-energy x-ray detection. The detectors were highly sensitive to soft x-rays and showed improved response compared to the commercial SiC UV photodiodes. Current-voltage characteristics at 475 K showed low leakage current revealing the possibility of high temperature operation. The high quality of the epi-layer was confirmed by x-ray diffraction and chemical etching. Thermally stimulated current measurements performed at 94–550 K revealed low density of deep levels which may cause charge trapping. No charge trapping on detectors' responsivity in the low x-ray energy was found. © 2012 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4742741>]

Silicon carbide (SiC), a wide band-gap semiconductor, has been recognized for high-power, high frequency, and high-temperature opto-electronics applications. Over the past decade, SiC has developed significantly in the area of high power electronics, making high-quality SiC material increasingly available for research and development and other commercial applications. This gained momentum to the development of SiC based ionizing radiation detectors, where a defect-free high purity single crystals and thick epitaxial layers are crucial for high resolution, high sensitivity, and low noise detectors of x-rays, gamma-rays, and low-energy ionizing radiation. Detectors based on 4H-SiC epitaxial layers with low level of impurities and defects can reliably detect any type of ionizing radiation at high radiation background at elevated temperatures and can be used in radiation doses as high as 22 MGy.<sup>1</sup> Diode-type detectors fabricated using SiC epitaxial layers<sup>2,3</sup> perform well in high-resolution detection of low penetration depth  $\alpha$ -radiation, whereas the resolution of the detectors based on bulk semi-insulating SiC grown by physical vapor transport (PVT) is not yet adequate presumably due to high density of defects and deep level centers,<sup>4</sup> implying that further quality improvement of these crystals is necessary.

In this work, we have evaluated the state-of-the-art n-type 4H-SiC epitaxial layers in terms of quality and electrical and defect properties. As there are no commercially available detector that is sensitive enough to soft x-rays in the sub-keV to 10 keV spectral range, our fabricated detectors showed significantly improved response of x-ray detection in such a low-energy range. We present results from bench-top electronic characterization, x-ray diffraction (XRD) rocking curve measurements, defect characterization by thermally stimulated current (TSC) spectroscopy, defect delineating chemical etching, and x-ray responsivity measurements (50 eV to 10 keV spectral range) performed using the X8A beam line at the National Synchrotron Light Source (NSLS) at Brook Haven National Laboratory (BNL).

We have used a 50  $\mu$ m thick n-type epitaxial layer grown on 2" diameter 4H-SiC (0001) wafer, which was highly doped with nitrogen and off-cut 8° towards the [11 $\bar{2}$ 0] direction. The net doping concentration of the epitaxial layer measured using high frequency (100 kHz) capacitance-voltage (C-V) method was found to be  $8 \times 10^{14} \text{ cm}^{-3}$ . The x-ray detectors were fabricated on  $8 \times 8 \text{ mm}^2$  substrates diced from the 2" diameter wafer by depositing 3.2 mm in diameter and of  $\sim 10 \text{ nm}$  in thickness Ni Schottky contacts on top of the epitaxial layers through the shadow mask using Quorum model Q150T sputter coater. Large Ni contact (approx.  $6 \times 6 \text{ mm}^2$ ) of  $\sim 100 \text{ nm}$  in thickness was deposited on the backside by the same means. The standard RCA cleaning of the substrates was used prior to contact deposition. No annealing was performed after the deposition of Ni contacts. The wire bonding technique has been developed in our laboratory for lead attachment without damaging the thin Ni contact. This technique deploys special type of silver paste rated for high temperature applications. The same type of the silver paste was used for mounting the chip on a PCB board. The photograph of the single pixel planar detector is shown in Fig. 1.

TSC measurements on the epitaxial layer were conducted in the temperature range 94–550 K in vacuum  $< 1 \times 10^{-4} \text{ Torr}$  at 4–15 K/min heat rates. The trap filling was achieved by illuminating the samples at 94 K using UVP model UVM-57 Handheld UV Lamp specified to produce 302 nm UV light. The detailed description of our TSC measurement set-up is available in our earlier work.<sup>5</sup> Current-voltage (I-V) characterization was performed at room and higher temperatures using Keithley 237 High Voltage Source Measure Unit.

In order to evaluate the density of crystallographic defects, defect delineating chemical etching was performed in molten KOH at 773 K for about 5 min. Threading edge, screw, and basal plane dislocation densities (BPDs) were assessed via etch pit density (EPD) evaluation using Nomarski optical microscope. X-ray diffraction rocking curves were acquired using double crystal diffractometer (model DSO-1, manufactured by Radicon Scientific Instruments

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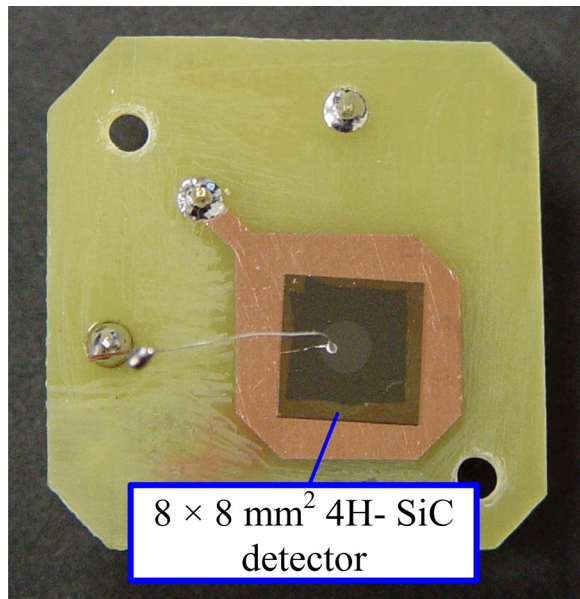


FIG. 1. Top-view photograph of 4H-SiC detector with 3.2 mm diameter Ni contact mounted on the PC board.

Ltd., Russia). We have used  $\text{CuK}_\alpha$  radiation and (0008) reflection in the rocking curve measurements.

The detectors fabricated at our laboratory at the University of South Carolina (USC), using n-type 4H-SiC epitaxial layers, were tested and evaluated at Los Alamos National Laboratory (LANL) for detecting low energy x-rays and compared to commercial off-the-shelf (COTS) SiC UV photodiode detectors (IFW JEC4). The measurements were performed at 20 V–250 V bias voltages using U3C and X8A beam line at the NSLS at BNL. The beam line provided monochromatic photon beams ranging from 50 to 6500 eV with intensities as high as  $10^{12}$  photons/second.

Figure 2 shows TSC spectra of the detector at 10 V and 3 V reverse bias both obtained at 15 K/min heat rate. The spectra are dominated by peak #1 with maximum temperature  $T_m = 108$  K and activation energy  $\sim 0.25$  eV estimated from Arrhenius plot (not shown). We associate this peak with relatively shallow acceptor-like levels near the valence band edge related to Al and/or B as well as to their complexes with intrinsic defects.<sup>6</sup> The intensity of this peak and peaks #2 and #4 clearly shows voltage dependence of the

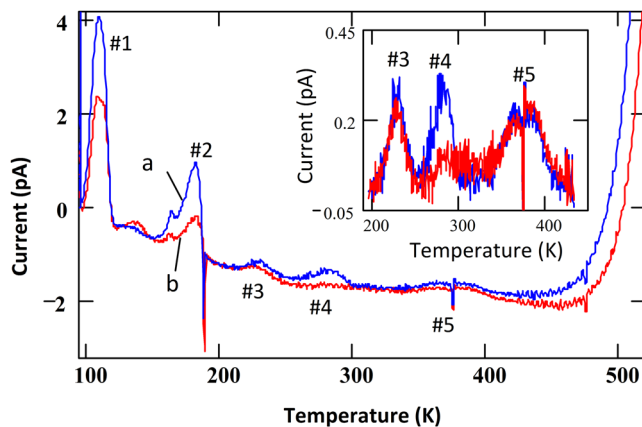


FIG. 2. TSC spectrum of n-type 4H-SiC epitaxial layer obtained at 10 V (a) and 3 V (b) reverse bias and 15 K/min heat rate.

peak intensity, implying that the deep level centers are evenly distributed in the depletion region. The intensity of peaks #3 and #5 do not show voltage dependence of the peak intensity as evidenced by inset in Fig. 2 showing these peaks after subtraction of the stray current from corresponding portions of the spectra in Fig. 2. Therefore these traps are probably located near the metal semiconductor interface and not in the bulk of the epitaxial layer. Assuming uniform distribution of deep level centers in the depletion region for peaks #1, #2, and #4 we have estimated their concentration ( $8 \times 10^{13} \text{ cm}^{-3}$ ,  $3 \times 10^{13} \text{ cm}^{-3}$ , and  $9 \times 10^{12} \text{ cm}^{-3}$ , respectively) using the formula

$$N_T = \frac{Q}{A} \sqrt{\frac{2N_d}{q\epsilon\epsilon_0(V_{bi} + V_a)}}, \quad (1)$$

where  $N_T$  is the trap concentration,  $Q$  is the total charge emitted by trap of a given TSC peak as determined by the area under the peak,  $A$  is the contact area,  $N_d$  is the net doping concentration,  $V_{bi}$  is the built-in voltage,  $V_a$  is the bias voltage,  $q$  is the electronic charge,  $\epsilon$  is the dielectric permittivity of SiC, and  $\epsilon_0$  is the dielectric permittivity of vacuum. We have used  $N_d$  determined from C-V characteristic at room temperature (RT) in Eq. (1), assuming  $N_T \ll N_d$ . Precise determination of activation energies for peaks #3–#5 using Arrhenius plot was not possible because the very weak TSC signal from these traps and low signal-to-noise ratio. Additionally peak #2 was always distorted by the negative spike, the origin of which is unclear at this time. Therefore for bulk traps #2 and #4 we performed trap identification using their maximum temperatures and results of our work<sup>5–7</sup> where we performed TSC studies of n-type and semi-insulating 4H-SiC samples using similar conditions. The peak #2 ( $T_m \sim 175$  K) may be due to HS1 (Ref. 8) and HH1 (Ref. 9) hole traps in the lower half of the bandgap. Trap #4 ( $T_m \sim 280$  K) peaked at temperatures typical to that of well-known  $Z_{1/2}$  center,<sup>8,10</sup> an electron trap associated with a vacancy-type defect.

The barrier height of  $\sim 1.8$  eV and  $\sim 1.6$  eV and the ideality factor  $\sim 1.06$  and  $\sim 1.35$  were determined from the I-V characteristics at RT and 475 K respectively. The dark current of the detector was below 1 nA at 200 V for both temperatures. The low leakage current at 475 K indicates the possibility of detector operation at high temperature. Note that the I-V characteristics were measured using dual stair sequence, and therefore each I-V characteristic in Figure 3 consists of two branches corresponding to ramping the voltage up and down. These branches did not coincide at RT resulting in a hysteresis, which we attribute to the influence of the deep level centers. One of the devices suffered breakdown at about 400 V at RT. Based on our previous studies<sup>11</sup> we expect it to occur at the device edges due to the low breakdown strength of the air and lack of surface passivation. Note that the breakdown voltage of the epilayer itself is  $\sim 8$  kV, implying on possibilities of further optimization. Development of proper edge termination and passivation is ongoing to extend the operating voltage of our detectors.

Quality of the epitaxial layers used for detector operation was assessed by defect delineating chemical etching in molten KOH and XRD rocking curve measurements. The rocking



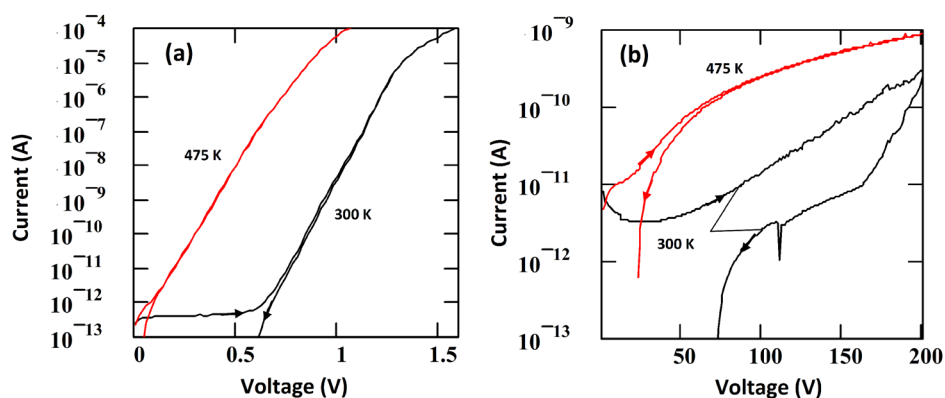


FIG. 3. Forward (a) and reverse (b) I-V characteristics at 300 K and 475 K for the 3.2 mm diameter detector on n-type 4H-SiC epitaxial layer.

curve acquired using (0008) reflection is shown in Figure 4. The full width at half maximum (FWHM) of the rocking curve is about 3.6 arc sec indicating high quality of the epitaxial layer. Results of defect delineating chemical etching revealed EPD of threading screw dislocations (TSDs)  $\sim 1.7 \times 10^3 \text{ cm}^{-2}$ , threading edge dislocations (TEDs)  $\sim 1 \times 10^4 \text{ cm}^{-2}$ , and BPDs  $\sim 70 \text{ cm}^{-2}$ . These dislocation densities are much lower than that reported earlier<sup>7</sup> reflecting improved quality of the epitaxial layer used in this study.

Synchrotron light sources such as the NSLS at BNL are highly suitable for probing the physical construction of photonic sensors and can also provide an absolute measurement of their responsivity. Figure 5 shows the responsivity of our detector and COTS SiC UV photodiode to soft x-ray energy ranges. Responsivity, given in units of A/MW, is measured by recording successive measures of photocurrent in a well-calibrated silicon sensor (with known responsivity) and in the sensor of interest. Ideally, the responsivity of a solid-state photon detector is determined solely by the inverse of the average electron-hole pair creation energy. However, dead layers and a limited active volume thickness lead to responsivity that varies greatly with photon energy. Further, edges are also apparent in the responsivity curve, arising from discrete atomic transitions. Edges associated with silicon and carbon are clearly observed in the data, providing a quantitative measure of the composition of the active and dead layers. The general feature of a steep decrease starting at 2–3 keV provides information on active layer thicknesses,

which is much higher in our detector. Therefore our sensor chip showed significantly improved response in the few keV range compared to COTS SiC UV photodiode. Our detector has shown much higher response in the low energy part of the spectra as well, which could be attributed to much thinner dead/blocking layers. Also, our detectors showed uniform response vs. energy from multiple positions on the detector chip as reported earlier.<sup>12</sup> Next, the detectors were tested for the charge trapping effects. Charge trapping can lead to space charge buildup, ultimately causing reduced charge collection efficiency. For high-energy photon beams used at the NSLS, which constantly deposit energy throughout the entire depth of the depletion zone, the effects of charge trapping may be inadvertently mitigated due to the de-trapping effect of the ionizing radiation. However, low-energy photons, which do not penetrate the full depth of the depletion zone, do not benefit from this de-trapping mechanism. Therefore, charge trapping should be more apparent in the low-energy response of the sensor. The strength of the electric field in the depleted region also plays an important role in charge collection efficiency in the presence of charge trapping, and the effect of charge trapping can be mitigated by applying stronger drift fields in the depleted zone. To search for any trapping effects at low photon energies, responsivity curves were collected for energies ranging from 50 eV to 1000 eV (Fig. 6). The data were statistically fit (solid lines in Fig. 6), which includes a metallization layer

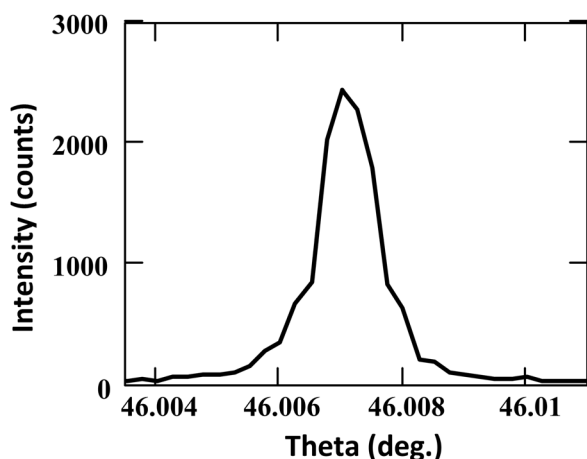


FIG. 4. Rocking curve ((0008) reflection) of the 4H-SiC epitaxial layer used for detector fabrication.

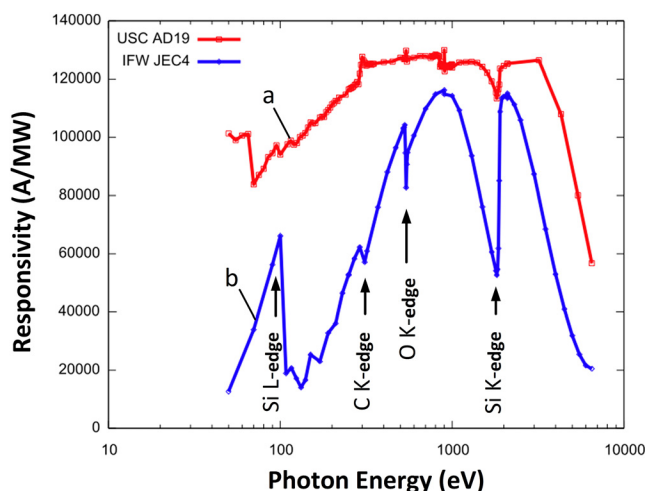


FIG. 5. Responsivity of the detectors on 4H-SiC n-type epitaxial layer (a) and COTS (IFW JEC4) SiC photodiode (b) to the low-energy x-rays.

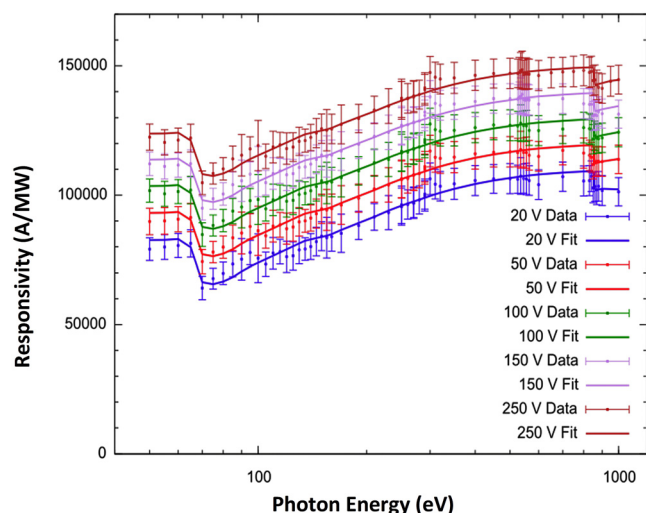


FIG. 6. Low-energy x-ray response for various bias voltages. Each subsequent data set is artificially offset in the vertical direction by +10 000 A/MW starting from an initial offset of -20 000 A/MW for the 20-V data set. The overlaid solid lines represent statistical fits in which the metallization thickness is allowed to freely vary.

and an active SiC layer as variable parameters. The data includes a scale factor to peak responsivity of the SiC photodiode, primarily sensitive to the charge carrier pair creation energy of the material and which was kept fixed to the value determined from the responsivity curve at 250 V bias. The width of the active layer  $W$  was calculated using the formula  $W = \sqrt{2\epsilon_0\epsilon V_a/qN_d}$ , and the metallization thickness had no statistically significant change of the bias voltage range from 20 V to 250 V. Figure 6 shows that for all bias voltages, the estimated data is in good agreement with the experimental data. Since estimated data does not account for any charge trapping, the agreement between calculated and experimental data is a good indication of the absence or negligible trapping effects in our detectors in the low energy x-ray region.

In conclusion, high sensitivity x-ray detectors in the low energy range (50 eV to 10 keV) on n-type 4H-SiC epitaxial

layers have been fabricated and evaluated. The fabricated detectors showed significantly improved response compared to the commercial COTS SiC UV photodiodes. The sensitivity to higher-energy photons ( $>3$  keV) was limited by the active volume thickness. TSC studies in wide temperature range of 94–550 K revealed the density of deep level centers in the order of  $10^{13} \text{ cm}^{-3}$ . No effects of charge trapping on detectors' responsivity were found. The high quality of the epitaxial layer was confirmed by XRD rocking curve measurements and defect delineating chemical etching. The epitaxial detectors exhibited low leakage current ( $<1$  nA) at 475 K revealing a great possibility of high temperature operation.

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