Silicon carbide diodes for neutron detection

José Coutinho\textsuperscript{a,∗}, Vitor J. B. Torres\textsuperscript{a}, Ivana Capan\textsuperscript{b}, Tomislav Brodar\textsuperscript{b}, Zoran Eresh\textsuperscript{b}, Robert Bernat\textsuperscript{b}, Vladimir Radulović\textsuperscript{c}, Klemen Ambrožič\textsuperscript{c}, Luka Snoj\textsuperscript{c,d}, Željko Pastuović\textsuperscript{e}, Adam Sarbutt\textsuperscript{e}, Takeshi Ohshima\textsuperscript{f}, Yuichi Yamazaki\textsuperscript{f}, Takahiro Makino\textsuperscript{f}

\textsuperscript{a}Department of Physics and I3N, University of Aveiro, 3810-193 Aveiro, Portugal
\textsuperscript{b}Ruđer Bošković Institute, Bijenička 54, 10000 Zagreb, Croatia
\textsuperscript{c}Jožef Stefan Institute, Jamova cesta 39, SI-1000 Ljubljana, Slovenia
\textsuperscript{d}University of Ljubljana, Faculty of Mathematics and Physics, Jadranska cesta 19, 1000 Ljubljana, Slovenia
\textsuperscript{e}Australian Nuclear Science and Technology Organisation, 1 New Illawarra Rd., Lucas Heights, NSW 2234, Australia
\textsuperscript{f}National Institutes for Quantum and Radiological Science and Technology, 1233 Watanuki, Takasaki Gunma 370-1292, Japan

Abstract

In the last two decades we have assisted to a rush towards finding a $^3$He-replacing technology capable of detecting neutrons emitted from fissile isotopes. The demand stems from applications like nuclear war-head screening or preventing illicit traffic of radiological materials. Semiconductor detectors stand among the stronger contenders, particularly those based on materials possessing a wide band gap like silicon carbide (SiC). We review the workings of SiC-based neutron detectors, along with several issues related to material properties, device fabrication and testing. The paper summarizes the experimental and theoretical work carried out within the E-SiCure project (\textit{Engineering Silicon Carbide for Border and Port Security}), co-funded by the NATO Science for Peace and Security Programme. The main goal was the development of technologies to support the fabrication of radiation-hard silicon carbide detectors of special nuclear materials. Among the achievements, we have the development of successful Schottky barrier based detectors and the identification of the main carrier life-time-limiting defects in the SiC active areas, either already present in pristine devices or introduced upon exposure to radiation fields. The physical processes involved in neutron detec-

\textsuperscript{∗}Corresponding author
Email address: jose.coutinho@ua.pt (José Coutinho)
tion are described. Material properties as well as issues related to epitaxial growth and device fabrication are addressed. The presence of defects in as-grown material, as well as those introduced by ionizing radiation are reported. We finally describe several experiments carried out at the Jozef Stefan Institute TRIGA Mark II reactor (Ljubljana, Slovenia), where a set of SiC-based neutron detectors were tested, some of which being equipped with a thermal neutron converter layer. We show that despite the existence of large room for improvement, Schottky barrier diodes based on state-of-the-art 4H-SiC are closing the gap regarding the sensitivity offered by gas-based and that of semiconductor detectors.

Keywords: Neutron detection, silicon carbide, radiation defects
1. Introduction

In the last two decades, we have witnessed a growing demand for devices capable of detecting neutron sources. Such a development is mostly explained by a shift of the end-usage, from niche (fundamental research or inspection of nuclear warhead limitation treatises [1, 2, 3]) to societal applications such as screening cargo at borders to prevent illicit traffic of radiological materials [4, 5].

Considering the limited stock of $^3$He available on Earth, semiconductor-based detectors spring up as a strong alternative with many advantages, particularly in terms of miniaturization and low operation bias, production yield and excellent gamma discrimination. Silicon carbide (SiC) is nowadays an established semiconductor sustaining a mature and growing industry of power electronics [6, 7, 8]. The motivation for leaving behind the traditional silicon technology emanates from a strong demand for devices with far greater power density, reliability, and overall performance (including cost). All of these are specifications that only a wide band-gap semiconductor like SiC with a large breakdown field, as well as exceptional thermal and mechanical stability, can offer.

Silicon carbide, in particular the electronic-grade 4H hexagonal phase (4H-SiC) [9, 10], has been proposed to be used for the fabrication of semiconductor-based ionizing radiation detectors [11, 12, 13], including neutrons [14, 15, 16, 17]. SiC-based detectors combine extreme radiation hardness with low leakage current, high signal to noise ratio, and excellent neutron/gamma discrimination for pulsed radiation, thus being a strong candidate to be used for particle detection under harsh conditions, including at high temperatures and radiation [18].

Besides SiC, diamond is also a strong contender for the fabrication of solid-state neutron detectors. Important advances in chemical vapor deposition (CVD) techniques, allowed the successful fabrication of synthetic-diamond neutron sensors [19, 20, 21, 22]. The advantages of diamond stem primarily from its large atomic displacement threshold energy (40-50 eV), which is substantially larger than that of SiC (20-35 eV). This property potentially makes diamond more radiation tolerant.

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detection has been reported \[23, 17, 24\]. For thermal neutrons, single crystal CVD diamond devices showed better neutron-gamma discrimination, although as pointed out by the authors, that was much owed to the use of a more effective thermal neutron conversion layer in the diamond structures, which led to a higher energy deposition within the detection volume \[24\]. Despite the larger atomic displacement threshold, diamond detectors showed polarization effects for neutron fluxes above \(10^9 \text{ cm}^{-2} \text{s}^{-1}\). Polarization of the structures arises from the creation of defects, where trapping/detrapping of carriers takes place, inducing transient space-charge fields which interfere with the signal from the ionizing radiation \[21\].

As for fast neutrons, both diamond and 4H-SiC detectors produced well characterized pulse high spectra with a well resolved \(^{12}\text{C}(n,\alpha)^{9}\text{Be}\) peak. The larger detection volume of the diamond device (with a capacitor structure) conferred a higher count rate than the SiC device \[17\].

A typical SiC-based detector has a structure of a Schottky barrier diode (SBD), like the one shown in Figure 1. Due to band alignment, a volume depleted of carriers is created at the semiconductor side of the junction, making the device very sensitive to the presence of electron-hole pairs generated upon illumination with above band-gap light or upon exposure to ionizing radiation. Since neutrons do not interact with valence electrons, their presence is deduced from detection of ionizing neutron reaction products, like gamma rays, alpha particles, tritons and larger ions. The device is operated under reverse bias, which increases the potential drop across the semiconductor and increases the depletion width. To limit the required operation voltage, the doping level of the substrate is usually two orders of magnitude higher than that of the epi-layer. Detailed specifications of SiC SBD detectors have been reported elsewhere by several authors using n-n^+ structures \[13, 14, 25, 26\]. Although more difficult to fabricate, p-n structures were also reported by Issa et al. \[27\].

The most probable interactions involving a fast neutron \((E_n > 10 \text{ MeV})\) impinging a SiC crystal involve elastic and inelastic recoil scattering events, \(^{12}\text{C}(n,n')^{12}\text{C}\) or \(^{28}\text{Si}(n,n')^{28}\text{Si}\) \[28, 29\], where some of the energy and momentum of the incident neutron is transferred to C and Si nuclei. If the hit is strong enough to knock out a crystalline atom from its site, the event can be recorded either as the permanent signa-
Figure 1: Design of a SiC Schottky barrier diode suitable for neutron detection. A converter layer covering the front contact is optional, and can substantially enhance the detection sensitivity to thermal neutrons due to the presence of a nuclide (such as $^{10}\text{B}$ or $^{6}\text{Li}$ isotopes) with high thermal cross section for $(n,\alpha)$, $(n,p)$, $(n,t)$ or similar reaction.

The sensitivity of a fast neutron detector can be further improved by adding a converter material over the front contact [26]. It consists of a layer rich on a nucleus with large scattering cross-section for fast neutrons. The resulting recoil ions are responsible for the generation of electron-hole pairs within the depletion region of the SBD. Among the most effective converter materials are those with high density of hydrogen because of its high scattering reaction cross-section. Another advantage of hydrogenous fast neutron converters consists on the large recoil penetration depth of H$^+$ into the semiconductor layer. Hydrogenous converter materials such as polyethylene show high conversion performance when compared to other materials [30]. Unfortunately, they usually cannot withstand high temperatures and/or harsh radiation environments.
Thermal and epithermal neutron detection can also be achieved via juxtaposition of a converter layer rich in isotopes with large cross-section for neutrons with energy in the range of $k_B T$ at room temperature (with $k_B$ representing the Boltzmann constant). Common choices are $^6\text{Li}$, $^{10}\text{B}$ or $^{235}\text{U}$. For a thermal neutron with kinetic energy $E_n = 0.0253$ eV (which corresponds to a velocity of 2200 m/s), their respective absorption cross-section is $\sigma_a = 938$, 3843 and 681 barn, way much larger than 0.17 and 0.00353 barn for $^{28}\text{Si}$ and $^{12}\text{C}$, respectively [31]. For $^6\text{Li}$ and $^{10}\text{B}$ the relevant reactions are respectively $^6\text{Li}(n,\alpha)t$ and $^{10}\text{B}(n,\alpha)^7\text{Li}$. Figure 1 displays a possible device architecture, where a $^{10}\text{B}$-rich front layer is employed [27, 32]. While the $^{10}\text{B}$ cross-section is about four times that of $^6\text{Li}$, it is important to note that the response of the detector depends on other factors, including the penetration depth of the reaction products into the depletion region. In that respect the alphas and tritons emitted by $^6\text{Li}$ with respective kinetic energy 2.05 MeV and 2.73 MeV, have the potential to generate more excitations than the alphas and $^7\text{Li}^+$ ions with 1.47 MeV and 0.84 MeV that result from the transmutation of $^{10}\text{B}$ [33].

Fission reactions involving $^{235}\text{U}$ split the uranium nucleus into two or exceptionally three energetic fission fragments and give rise to the emission of secondary neutrons and gamma radiation. Unfortunately, the interaction of heavy fission fragments with the detection material is also responsible for considerable damage inflicted on the device, particularly for fluences above $10^{13}$ n/cm$^2$, dramatically decreasing the observed count rate [15].

The characterization of crystalline defects, either produced during operation or those already present in as-grown material, is of paramount importance. As pointed out by several authors [34, 35], the presence of a small group of defects in the SiC active layer can ultimately determine the detector performance. Defects perturb the crystalline periodicity by introducing potential wells where charge carriers can be trapped. Weak perturbations induce shallow states, where electrons and holes spend little time before being emitted back to the valence or conduction band. On the other hand, strong perturbations produce deep states, from which carriers escape with more difficulty [36]. Shallow and deep states in SiC can hold carriers with binding energies of the order of 0.1 eV and 1 eV, respectively. The ability of a defect to trap a carrier is essentially
determined by its capture cross section, and eventually by the existence of a significant capture barrier [37].

When a semiconductor contains trapping defects, i.e. locations that interact with a flux of carriers via trapping and detrapping events, the collection of the charge produced by the incident radiation is delayed and not fully accounted for during the signal integration time. Moreover, after trapping a carrier, defects may become strongly attractive to carriers of opposite charge, eventually leading to recombination via multi-phonon emission [37]. The consequence is the dissipation of the detector signal through heating. It is therefore clear that the characterization of defects in SiC, either present in as grown material, introduced during technological processes, or those created by ionizing radiation, is a crucial step towards improving radiation hardness and performance of SiC-based detectors.

The carrier life-time is an important property with direct impact on detection performance [38]. One promising way to improve carrier life-time is to limit the presence of recombination defects and impurities via defect engineering. Important breakthroughs were achieved by the groups of Kimoto [39] and Svensson [40], who found that high-temperature (∼ 1200 °C) oxidation followed by removal of the oxide layer leaves a SiC top-layer ∼ 50 μm deep, with the life-time being improved by at least a factor of two with respect to the as-grown figure. The authors proposed that self-interstitials, injected into the SiC during oxidation, were able to annihilate carbon vacancies (V_C defects), decreasing their concentration to a level below 10^{11} cm^{-3} [39, 40]. Carbon vacancies are even present in state-of-the-art epitaxial 4H-SiC. The defect has a rather low formation energy of 4.8 eV [41], forms during high-temperature annealing (without irradiation), and it is the main life-time-limiting defect in electronic-grade SiC. Other promising techniques to control the concentration of V_C defects include annealing of SiC encapsulated in a carbon-rich pyrolyzed resist film [41, 42], or ion-implantation [43, 44, 45].

Defects in SiC and particularly in 4H-SiC, have been extensively studied using several techniques, among which we single out electron paramagnetic resonance (EPR), photoluminescence (PL) and deep level transient spectroscopy (DLTS) [9, 10]. The latter is particularly powerful regarding the assessment of the impact of defects on de-
tector performance. DLTS is a junction spectroscopy method which gives us access to the depth of traps (with respect to the semiconductor band edges) and capture cross-sections for carriers. Details about DLTS and related techniques such as Laplace-DLTS (L-DLTS) have been extensively discussed elsewhere [46, 47, 36].

Theory has also played a central role in the identification of defects in SiC [9, 48, 49], in particular electronic structure methods based on density functional theory [50]. Density functional software packages are common tools in modern laboratories, with which one can simulate an electronic-scale picture of defects in solids, surfaces and nanostructures [51, 52]. Many observables can be calculated and directly compared to measured data, including $g$-tensors [53, 54], absorption and luminescence line shapes [55], barriers and rates for thermally stimulated emission and capture of carriers [56], vibrational spectra [57, 58], migration and reorientation [59, 60], stress-response of spectroscopic signals [61], among many others.

The general requirements of neutron detectors for monitoring applications can be divided into two main categories, the first related to the active components themselves and the second related to the system implementation. In the first category we have the detection efficiency (essentially the fraction of detected neutrons, usually expressed as a percentage), background discrimination against gammas, response linearity, long-term stability and radiation hardness. Requirements in the second category include the temperature stability or operating temperature range, environmental factors like the tolerance to mechanical shock, need for data acquisition/storage, maintenance, etc.

Requirements related to the active components can be addressed by the selection of the underlying physics of detection (and subsequently by detector design, including materials choice, optimizing geometry, or tailoring the acquisition system properties). In this respect, it will be shown that carefully designed SiC detector prototypes can offer high detection sensitivity, already matching current BF$_3$ detector technology [62], and approaching $^3$He detector technology. Additional specs offered are linear response to the incident neutron flux and excellent degree of gamma discrimination (practically 100% sensitivity to neutrons) [63]. The later property is critical to isolate the neutron signal from the often accompanying gamma and X-ray radiation fields. This is possible with materials like SiC and diamond thanks to the low atomic number of their
constituents [14] [64] [23]. It is also noted that neutron-gamma discrimination in SiC SBD detectors can be tuned by changing the working bias [24].

Radiation hardness of SiC is also addressed in this review. It will be shown that defects, i.e. radiation-induced degradation of the detectors, only occurs for epithermal / fast neutron fluence levels of the order of $10^{11}$ n/cm$^2$ [65] [66], which is extremely high in the context of border and port security monitoring. Macroscopic radiation hardness or the long-term stability of these detectors are not covered.

Requirements related to the system implementation are addressed by design of structural components (including housing, thermal insulation or temperature control, mechanical isolation and damping) and the acquisition system. For border and port monitoring it is advantageous if detectors and associated electronics are robust, simple to operate/maintain, and operable in a wide temperature range. The mechanics of SiC itself, being an extremely resistant ceramic being used in a variety of harsh applications, combined with the wide electronic bandgap and ability to be doped with both n- and p-type dopants, are key advantages in view of these requirements. Regarding the acquisition systems, for a single SiC-based detector they are very similar to those of gas-based ionization detectors. However, in the context of border and port monitoring, large arrays of SiC detectors would be needed, each detector registering a separate signal, needing to be digitized, processed and combined. This presents a technological challenge, which reminds us the complexity of detectors developed in the context of high-energy physics experiments. However, this will be largely mitigated with the rapid development of accessible, open source, low cost and highly complex electronics, which are leveraging advances in a multitude of different fields, including nuclear and aerospace.

In the following sections, we will describe a series of experiments and calculations involved in the design, fabrication and characterization of 4H-SiC thermal neutron detectors. We start Section 2 describing the properties of SiC materials. We proceed to Section 3 where we detail the growth of the SiC layers and fabrication of the Schottky junctions. In Section 4 we report on the characterization of the most harmful defects present in as-grown and irradiated material. Particular attention is given to the $V_C$ defect. In Section 5 we describe a prototype detection system, followed by the results
from field-testing. Finally, conclusions and an outlook are drawn in Section 6.

2. SiC properties

Silicon carbide is perhaps the best example of a compound that shows polytypism. This property consists on the formation of distinct polymorphs (different crystalline structures with a common stoichiometry) coherently stacked along a crystalline direction. In SiC, the stacking occurs along the \( \langle 0001 \rangle \) direction of the hexagonal close-packed lattice. According to Figure 2(a), three possible sites are available, namely A, B and C (shown as black, gray and white circles, respectively). The stacking is made with \( \langle 0001 \rangle \)-aligned Si-C dimers, so that each atomic bilayer is of type A, B or C. Although there are in principle infinite combinations of periodic stacking sequences, only a few are grown with acceptable quality, namely (AB), (ABC), (ABCB) and (AB-CACB). According to Ramsdell’s notation [67], these polytypes are referred to as 2H, 3C, 4H and 6H, respectively, indicating the number of SiC bilayers per unit cell and the lattice system (H - hexagonal, C - cubic, R - rhombohedral).

It is also common to distinguish Si-C dimers by their site-symmetry as perceivable by the arrangement of their first neighbors [68]. For instance, the A,h-labeled Si-C dimer of stacking type A at the bottom of Figure 2(b) has three Si and three C neighbors whose locations are equivalent by symmetry with respect a (0001) mirror plane crossing the Si-C bond. In 2H-SiC (wurtzite structure) all Si-C dimers show this arrangement, and because of that they are labeled with an “h” (standing for hexagonal site). The same happens with Si-C dimers of stacking type C in Figure 2(b). On the other hand, Si-C dimers of stacking type B have neighbors whose sites do not map to each other upon \( \{0001\} \) reflection. Instead, their neighboring sites transform with inversion symmetry at the center of the bond. This is analogous to the case of cubic 3C-SiC (zincblende structure), where all Si-C bilayers show this arrangement. Hence, in the case of hexagonal polytypes, these sites are labelled with the letter “k”, which

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1We are assuming an ideal crystalline structure with all atoms forming identical and perfect tetrahedral bonds with their neighbors.
Figure 2: Crystalline properties of SiC. (a) Possible stacking sites (A, B or C) for Si-C dimers shown as black, gray and white circles, respectively. (b) Crystal structure of 4H-SiC illustrating the (ABCB) stacking sequence combined with the site-symmetry of atoms: pseudo-cubic (A,k and B,k) and hexagonal (A,h and B,h). Carbon and silicon atoms are colored gray and white, respectively. (c) High-symmetry crystallographic planes (within parentheses) and directions (within square brackets) using the four-index Miller-Bravais and Weber notations, respectively.
stands for cubic (or more appropriately pseudo-cubic, considering that the cubic-like symmetry is lost if we consider atomic shells beyond the first neighbors).

For the sake of reference, we also depict in Figure 2(c) a few high symmetry planes and directions using the four-index Miller-Bravais and Weber notations, respectively, which are commonly used for hexagonal crystal systems [69]. These can be related to the three-index Miller notation. The conversion of an \((hkl)\) plane to the Miller-Bravais notation is straightforward, essentially involving the addition of a third basal index (which is linearly dependent on \(h\) and \(k\)),

\[
(hkl) \equiv (hkil), \text{ with } h + k + i = 0. \tag{1}
\]

Although being redundant, the index \(i\) makes index permutation symmetries more clear. For instance, as depicted in Figure 2(c), planes \((1010)\) and \((1100)\) are symmetrically equivalent, and that would not be apparent if they were represented within the Miller scheme as \((100)\) and \((110)\), respectively. For crystallographic directions, an analogous four-axes extension is available, known as Weber notation, where \([UVTW]\) relates to a three-index \([uvw]\) counterpart as

\[
U = (2u - v)/3 \tag{2}
\]
\[
V = (2v - u)/3 \tag{3}
\]
\[
T = -(u + v)/3 \tag{4}
\]
\[
W = w, \tag{5}
\]

where \(U + V + T = 0\) is again verified, and the Weber indices of the direction perpendicular to a lattice plane are the same as the Bravais-Miller indices of that plane. Like in the Miller-Bravais notation for planes, the Weber notation exhibits all permutation symmetries among equivalent directions. This is shown in Figure 2(c), where the equivalence of \([2\bar{1}10]\) and \([\bar{1}\bar{1}20]\) is much more evident than if they were represented as \([100]\) and \([\bar{1}\bar{1}0]\), respectively.

We can think of all possible polytypes as ranging from \(2H\) (purely hexagonal) up
Figure 3: Variation of the band gap of SiC polytypes as a function of their hexagonal fraction. Both experimental (squares) and theoretical estimates within several approximations, namely the generalized gradient approximation (PBE), hybrid density functional approximation with screened exact exchange (HSE06), and many-body perturbation theory within $GW$, are shown.

to 3C (purely cubic), with other structures representing intermediate cases with partial hexagonality. $4H$- and $6H$- for instance, have 1/2 and 1/3 fractions of hexagonal bilayers (per unit cell). Interestingly, the band gap width of SiC increases monotonically with the hexagonality fraction [70]. At cryogenic temperatures, the experimental band gap widths are $E_g = 2.39$ eV, 3.02 eV, 3.26 eV and 3.33 eV for 3C-, 6H-, 4H- and 2H-SiC, respectively [9, 71]. These values are plotted in Figure 3 (squares), along with analogous quantities obtained from first-principles employing different approximations to the exchange-correlation interactions between electrons. These include the generalized gradient approximation (PBE, circles), hybrid density functional approximation involving a mix of local and non-local screened exchange contributions (HSE06, triangles), and many-body perturbation theory within the Green’s function method with screened potential ($GW$, diamonds).

Despite the relative offsets with respect to the observed $E_g$ values, theory describes rather well the variation with the hexagonal fraction. Although not currently clear, the
reasoning for such variation is possibly connected with an increase in the ionicity of the phases with higher hexagonal fraction due to an increase of the internal crystal field.

The $E_g$ values obtained within the generalized gradient approximation (according the Perdew, Burke and Ernzerhof, PBE [72]) underestimate the experiments by about 50%. This is a well known insufficiency of this method, and it is mostly attributed to self-interaction errors. These effects are accounted for by many-body perturbation calculations [73] ($GW$, diamonds), which removes self-interactions in the electronic correlation, and that leads to $E_g$ values already very close to the measured figures. We can also note from Figure 3 than the $GW$ results invariably underestimate the experimental data by up to 0.2 eV. This is explained by the slightly over-delocalized one-electron states used, which were taken from a density functional calculation within the generalized gradient approximation.

Figure 3 also shows the band structure obtained using hybrid density functional theory. This method provides $E_g$ values comparable to those obtained by $GW$. Essentially, hybrid density functional theory mixes a portion of exact exchange (in the spirit of the Hartree-Fock method) using the Kohn-Sham orbitals. Electron correlation interactions are still addressed using a local or semi-local method (such as PBE). Despite having to evaluate a four-center integral involving the Kohn-Sham orbitals, hybrid density functional methods are still much lighter than $GW$, which accounts for the many-body electron-electron interactions via screening of the exchange interactions using a frequency-dependent microscopic dielectric matrix.

The specific hybrid density functional employed, HSE06 (firstly proposed by Heyd, Scuseria, and Ernzerhof [74] and subsequently improved by Krukau et al. [75] in 2006), numerically screens the weak long-range exchange contributions, making the method more efficient and as accurate as bare hybrid functionals.

Figure 4 shows the first Brillouin zone for a hexagonal crystal, along with the electronic band structure of $4H$-SiC calculated using many-body perturbation theory ($GW$) and hybrid density functional theory (HSE06). Importantly, both methods show quite similar band structure across the entire Brillouin zone. The origin of energy was set at the top of the valence band in both calculations. As already referred above, the differ-
Figure 4: (a) First Brillouin zone of a hexagonal lattice along with reciprocal-space segments between several high-symmetry \( k \)-points. (b) Electronic band structure of 4H-SiC based on quasi-particle energies from many-body perturbation calculations \((GW)\) and based on the Kohn-Sham energies from hybrid density functional calculations \((\text{HSE06})\).

The difference between the band gap widths in both calculations is only 0.1 eV \( (E_g = 3.04 \text{ eV} \) and 3.17 eV from GW and HSE06 theory levels, respectively). The valence band top and conduction band bottom states occur at \( k = \Gamma \) and \( k = M \) (along \( \langle 2\bar{1}10 \rangle \) in reciprocal space), respectively. The similarity of the results from both methods suggest that electronic structure calculations of defects that introduce deep traps into SiC can be accurately calculated using hybrid density functional methods \([76]\).

We end this section with a summary of some physical properties of the most common SiC polytypes, which are reported in Table 1.

3. Material growth and device fabrication

Active layers of SiC electronic devices are grown over a wafer made of bulk material which provides mechanical support. The most common method to grow bulk SiC is seeded sublimation (also known as modified Lely method) \([80]\). Accordingly, SiC powder is RF-heated in a graphite crucible to about 2500 °C, and upon sublimation, Si\(_2\)C and SiC\(_2\) molecules are deposited on a SiC seed crystal, which is located near the lid of the crucible, and kept at a slightly lower temperature to promote condensation (see Chapter 4 of Ref. \([10]\) and references therein). Currently, several SiC manufacturers
Table 1: Some physical properties of SiC polytypes as function of their hexagonal fraction (HF): Lattice parameters at room temperature (for the sake of comparison with the hexagonal phases, $a$ and $c/a$ for 3C-SiC are reported considering $c/a = a_0 \sqrt{2}/2$, where $a_0$ is the conventional cubic lattice parameter) [77], bulk modulus ($B$) [77], static dielectric constant ($\epsilon_0$) values from the 6H-SiC polytype are normally used for 4H-SiC. For 2H-SiC only an orientationally average value is available) [77], electronic band gap width ($E_g$) [78], electron and hole mobility ($\mu_{eh}$) [79], and breakdown field ($E_B$). A doping concentration of $10^{16}$ cm$^{-3}$ is assumed when applicable.

| Property | 3C-SiC | 6H-SiC | 4H-SiC | 2H-SiC |
|----------|--------|--------|--------|--------|
| Space group | $F\bar{4}3m$ | $P6_3mc$ | $P6_3mc$ | $P6_3mc$ |
| HF | 0 | 1/3 | 1/2 | 1 |
| $a$ (Å) | 3.0827 | 3.0806 | 3.0798 | 3.0763 |
| $c/a$ | 2.178 | 4.907 | 3.262 | 1.641 |
| $E_g$ (eV) | 2.39 | 3.02 | 3.26 | 3.33 |
| $\epsilon_0$ | 9.72 | 9.66 | 10.03 | 10.0 |
| $\epsilon_0$ |  |  |  |  |
| $B$ (GPa) | 230 | 230-234 | 217 | 223 |
| $\mu_{e,\perp}$ (cm$^2$/Vs) | 750 | 360 | 800 |  |
| $\mu_{e,\parallel}$ (cm$^2$/Vs) | 97 | 97 | 880 |  |
| $\mu_{h}$ (cm$^2$/Vs) | 40 | 90 | 115 |  |
| $E_B$ (MV/cm) | ~ 4 | ~ 3 | ~ 3 |  |
supply single crystal wafers produced by seeded sublimation with a diameter as large as 6-inch.

Active layers in devices are produced by means of homoepitaxial growth using CVD, allowing polytype replication and both p- and n-type doping. This is achieved by using the so-called step-flow growth and by controlling the C/Si ratio, respectively. Silane (SiH$_4$) and propane (C$_3$H$_8$) are common precursor gases, while hydrogen (H$_2$), sometimes mixed with argon (Ar), is used as carrier gas. The typical growth temperature is in the range 1600-1650 °C and the growth rate is around 100 µm/h (about 10 times slower than substrate growth by seeded sublimation). Developments in CVD growth of SiC by Ito and co-workers [81] achieved simultaneous high growth rate (up to 250 µm/h), large-area uniformity and doping homogeneity.

A schematic diagram of a CVD reactor is depicted in Figure 5. The design incorporates the essential features of the reactor used to grow the epi-SiC layers of the
Schottky diodes which will be described below. It comprises a vertical hot-wall reactor with downward gas flow, with graphite walls heated by induction coils, while heating of the rotating susceptor is achieved via internal radiation.

The reactor setup possesses a gas injector allowing reactants to be supplied directly onto the substrate, thus helping to achieve high growth rates. An H₂-etching step preceding the actual growth is performed at about 1400 °C. The purpose of the in-situ etching is two-fold, namely (i) pull off a few top layers of the substrate to remove subsurface damage and (ii) obtain a regular stepped surface which is essential for the replication of the underlying substrate polytype (thus the name step-controlled epitaxy) [82]. The particular success of 4H-SiC homoepitaxy in terms of the resulting SBD quality, paved its way to become the material of choice for power device applications.

A growth rate as high as 250 µm/h has been achieved at a SiH₄/H₂ ratio of 0.005 (C/Si ratio fixed to 1.0) [81]. For higher ratios (and consequently gas flow rates), the epi-layer surface became rough due to formation of 3C-SiC domains. Large-area uniformity has been optimized by allowing a lateral displacement of the gas injection with respect to the center of the reactor (see Figure 5). This enhances the effect of the susceptor rotation, and leads to uniform growth rates and doping concentration along the radial direction of the substrate [81]. The resulting material shows good optical and electrical specifications. Photoluminescence spectroscopy shows that free-exciton recombination stands as the main radiative decay channel. On the other hand, DLTS indicates the presence of three deep traps, the two most prominent (commonly referred to as Z₁/₂ and EH₀/7) occurring with a concentration below 10¹³ cm⁻³.

The SiC detectors which will be reported in the sections below, consisted of Schottky barrier diodes based on n-type 4H-SiC. A Schottky (rectifying) junction between a metal and a n-type semiconductor is formed when the work function of the metal exceeds the electron affinity of the semiconductor (φₘ > χₛ), while an Ohmic contact is formed when (φₘ ≤ χₛ). Because 4H-SiC has a relatively low electron affinity, most metals form Schottky barriers. The barrier height is given by φₙ = φₘ − χₛ and depends whether it is formed on the C-face or Si-face surface (φₜₐ₃ or χₛ). For the case of a Ni contact, these were measured as eφₜₐ₃ = 1.87 eV and eφₜₛ = 1.69 eV, respectively [83]. Ohmic contacts on n-type 4H-SiC are often made by annealing a layer of
Figure 6: Diagram with defect states within the band gap representing (a) a generation process via a deep state in a depletion region, (b) electron trapping in n-type, (c) hole trapping in p-type, and (d) recombination of carriers. Symbols \( e_n \) and \( C_n \) denote emission and capture processes for electrons and holes (subscripted \( n \) and \( p \), respectively).

the same metal used for the Schottky barrier. The thermal treatment has the effect of creating a thin silicide with a reduced barrier height \([84]\).

In the present work we used n-type silicon carbide SBDs, fabricated using nitrogen-doped ([N] in the range \((4.7-9.1) \times 10^{14} \text{ cm}^{-3}\)) \(4H\)-SiC epi-layers with a thickness in the range 25-170 \(\mu\text{m}\), grown on \(n^+\)-type wafers. The Schottky barrier was formed by evaporation of nickel through a metal mask with patterned squared apertures of 1 mm \(\times\) 1 mm, while Ohmic contacts were formed by nickel sintering at 950 °C in Ar atmosphere on the back side of the silicon carbide substrate \([85]\).

4. Defect characterization

Depending on their affinity for electrons and holes, deep level defects can act as trapping, recombination or generation centers. Deep level defects severely affect the performance of devices and thus are followed with interest by the semiconductor scientific and industrial communities. Their characterization is therefore crucial for future improvement of the radiation hardness and extending the integrity of semiconductor detectors.
The technique of choice for deep level defect characterization is DLTS. The method has played a prominent role towards our understanding of electrically active defects in semiconductors, combining high sensitivity with reasonable energy and spatial resolution. In DLTS, a steady reverse voltage applied to a junction (Schottky or p-n) is perturbed by a pulse bias, which has the effect of injecting majority carriers into the space-charge volume thus filling available deep traps (provided that the filling pulse is long enough). Upon removal of the pulse bias, majority carriers are emitted back to the crystalline states and freed from the traps, thus restoring the width of the depletion region. The realm of DLTS lies on following the rates of filling and emptying of the defect traps by measuring the capacitance transients of the sample diode \[46, 36\].

Figure 6 represents the main carrier emission/capture events that can be measured by DLTS. Analysis of the kinetics underpinning these processes is usually based on Schokley-Read-Hall (SRH) statistics \[87, 37\]. For the case of an n-type semiconductor, the rate of capture of a flux of electrons by a deep trap possessing a characteristic

\[
\Delta E_1 = 0.59 \text{ eV}
\]
\[
A_1 = 1.7 \times 10^7 \text{ s}^{-1} \text{K}^{-2}
\]
\[
\Delta C_1 = 0.0065 \text{ pF}
\]
\[
\Delta E_2 = 0.67 \text{ eV}
\]
\[
A_2 = 2.1 \times 10^7 \text{ s}^{-1} \text{K}^{-2}
\]
\[
\Delta C_2 = 0.031 \text{ pF}
\]

Figure 7: Conventional DLTS spectrum (data points) obtained for an as-grown n-type 4H-SiC SBD. Reverse bias, pulse voltage, and width were \(V_r = -10 \text{ V}, V_p = 0 \text{ V}, \) and \(t_p = 1 \text{ ms}\), respectively. The solid line is the simulated DLTS spectrum. Reproduced from Ref. \[86\] with the permission of AIP Publishing.
electron capture cross-section $\sigma_n$ is

$$ C_n = \sigma_n (v_n) n, \quad (6) $$

where $n$ is the free-electron density traveling at an average thermal velocity $\langle v_n \rangle = (3 k_B T / m^*)^{1/2}$ and $m^*$ is the free-electron effective mass. The capture cross section describes a thermally-activated capture process [37] and has a temperature-dependence of

$$ \sigma_n (T) = \sigma_{\infty} \exp (-\Delta E_{\sigma} / k_B T). \quad (7) $$

Equation (7) depends on the so called direct capture cross-section $\sigma_{\infty}$ and capture barrier $\Delta E_{\sigma}$, and these quantities are usually measured by following the capture rate and varying the duration of the filling pulse [36]. It is noted that an analogous set of equations could be written for the capture of holes.

Immediately after the filling pulse, the diode becomes again reverse biased and electron emission becomes dominant over capture. The rate of electron emission to the conduction band is then

$$ e_n = \sigma_{\infty} g (v_n) N_c \exp (-\Delta E_{\text{na}} / k_B T), \quad (8) $$

where $g$ accounts for the degeneracy ratio between states before and after electron emission, $N_c$ is the effective density of states in the conduction band, and $\Delta E_{\text{na}}$ is an activation energy for electron emission from the trap to the conduction band bottom.

The standard procedure of a DLTS measurement is to find the activation energy for carrier emission (from a trap) performing a sequence of isothermal measurements at different temperatures. This is done by monitoring the capacitance of the diode at the selected temperature after filling the trap, which should vary as

$$ \Delta C(t) = \Delta C_{\text{max}} \left[ 1 - \exp (-e_n t) \right], \quad (9) $$

and determining the emission rate $e_n$ for that particular temperature. Because the $\langle v_n \rangle N_c$ product has a $T^2$-dependence, DLTS emission data is usually cast as a log plot of $e_n / T^2$.
against $1/k_B T$, and therefore ready to be fitted against an Arrhenius straight line. From the fit, the intercept of the plot with the log($e_n/T^2$)-axis at $1/k_B T = 0$ is identified as the apparent capture cross section, whereas the slope is the activation energy for electron emission, $\Delta E_{na} = \Delta E_t + \Delta E_{\sigma}$, allowing us to extract $\Delta E_t$, i.e. the actual location of the trap with respect to the conduction band minimum [36, 37, 47]. It is noted that due to large error bars, the apparent capture cross-section can differ considerably from the cross-section directly measured from the filling kinetics ($\sigma_{n\infty}$).

Despite the success of DLTS in the identification of deep traps in semiconductors, this technique faces severe difficulties in discriminating traps with close emission/capture kinetics. This is the case of impurities under the effect of slightly different fields, for instance, impurities in alloys with different neighbors (strain fields), or more importantly in the present context, for defects located at hexagonal or pseudo-cubic sites of the 4H-SiC lattice. To overcome this problem, the most successful method has been Laplace-DLTS [47]. This method is based on the acquisition and averaging of the emission transient at a constant temperature, followed by an inverse Laplace transform of the signal. This allows for the extraction of individual contributions within the acquired data, and leads to an energy resolution of one order of magnitude higher than conventional DLTS.

4.1. As-grown defects in epitaxial 4H-SiC

The main life-time limiting defect (also referred to as “life-time killer”) in as-grown 4H-SiC material is the carbon vacancy. The defect is detected by DLTS as a conspicuous peak around room temperature, and it was earlier designated by EH2 or more commonly by Z$_{1/2}$ [88, 89]. A typical Z$_{1/2}$ spectrum consists of an asymmetric peak as shown in Figure 7 (see for instance Ref. 86). It is the dominant trap in as-grown epitaxial 4H-SiC, showing up with concentrations in the range $10^{12}$-$10^{13}$ cm$^{-3}$. In order to account for the peak asymmetry, the figure also includes a simulated DLTS signal (solid line), which was fitted to the data in order to reproduce a superposition of two close peaks. The resulting activation energies for electron emission are $\Delta E_1 = 0.59$ eV and $\Delta E_2 = 0.67$ eV, respectively (see legend in the figure). The Z$_{1/2}$ defect is rather stable, sustaining thermal treatments of up to 1400 °C [90].
Figure 8: Arrhenius plots of electron emission rates for $Z_1(=/0)$, $Z_2(=/0)$, $Z_1(=0)$ and $Z_2(=0)$ transitions in $4H$-SiC obtained by Laplace-DLTS measurements. Activation energies for electron emission ($\Delta E_a$) are also shown for each peak. Reproduced from Ref. [86] with the permission of AIP Publishing.

Hemmingsson et al. [91] assigned $Z_{1/2}$ to the superposition of two very close DLTS signals, each arising from a sequential emission of two electrons, thus showing negative-$U$ ordering of levels. More recently, their connection with electron paramagnetic resonance data, allowed the unambiguous identification of $Z_{1/2}$ with the carbon vacancy [92].

Recently, Capan and co-workers were able to clarify the carrier emission and capture dynamics of $Z_{1/2}$ by applying conventional and Laplace-DLTS in combination with first-principles modeling [86, 65]. The broad $Z_{1/2}$ peak was after all found to comprise a total of four distinct emission processes [86, 65], consisting of single electron emissions ($=0$) and double electron emissions ($=0$), each involving the carbon vacancy located on the k and h sites of the $4H$-SiC lattice. These are often labeled $V_C(k)$ and $V_C(h)$ defects, respectively. The resulting Arrhenius plots from the high-resolution Laplace-DLTS method are shown in Figure 8.

Based on a comparison between the relative depth of the traps with the respective
Figure 9: (Top) Possible structures of the carbon vacancy in 4H-SiC for several charge states. The [0001] axis is perpendicular to the plane of the figure. Si atoms are shown as white circles. The missing C-atom lies below the central Si atom. Thick lines indicate the formation of reconstructed bonds between Si second neighbors. (Bottom) configuration coordinate diagram for neutral, negative, and double negative $V_C$ defects in n-type 4H-SiC. Transformation barriers are indicated next to the arrows. All energies are in eV. Reproduced from Ref. 86, with the permission of AIP Publishing.
calculations, and a comparison between the relative amplitudes of the emission signals with the calculated relative stability of $V_C(k)$ and $V_C(h)$, it was possible to assign $V_C(k)$ and $V_C(h)$ to $Z_2$ and $Z_1$ signals, respectively. Both $Z_1$ and $Z_2$ showed a negative-$U$ ordering of the acceptor levels. $Z_2$ had the larger correlation energy ($U = -0.23$ eV) with levels at $E(-/0) = E_c - 0.41$ eV and $E(=/-) = E_c - 0.64$ eV, while $Z_1$ had levels separated by only $U = -0.11$ eV, and they were found at $E(-/0) = E_c - 0.48$ eV and $E(=/-) = E_c - 0.59$ eV [86]. The capture kinetics was also investigated for $Z_{1/2}$. Only $Z_2(=/-)$ had a measurable capture barrier (0.03 eV), making the activation energy for the doubly charged $Z_2$ emission 0.67 eV (c.f. Figure 8). The capture mechanisms were found to involve the transformation between different structures of the vacancy. This conclusion is in line with the negative-$U$ character of the defect, which must involve strong relaxations upon changing the charge state [93].

For the sake of example, let us look at Figure 9, in particular at the configuration coordinate diagram of vacancies located at the pseudo-cubic sites, $V_C(k)$. Before the filling pulse is applied, the sample diode is reverse biased and all vacancies are in the neutral charge state. Upon injection of majority carriers, electrons are captured by $V_C^0(k,B)$ and their structure quickly transforms to $V_C^-(k,D)$. The binding energy of the electron to the defect (trap depth) was measured as 0.41 eV [86] and calculated as 0.61 eV [76]. Due to the negative-$U$ ordering of levels, $V_C^-(k,D)$ is more eager for electrons than $V_C^0(k,B)$ and if free electrons are still available in the conduction band, negatively charged defects will actually capture a second electron at a rate faster than that of the first capture. The binding energy of the second electron captured was measured as 0.64 eV (calculated as 0.64 eV [76]). Analogous results were found for the vacancy at the hexagonal site.

The presence of carbon vacancies in SiC seems unavoidable. The reason stems from its rather low formation energy. This quantity was estimated by first-principles within hybrid density functional theory [94, 76] as 5.0 eV and 4.4 eV under C-rich and Si-rich growth conditions, respectively. The C-rich results agree very well with a formation enthalpy of 4.8-5.0 eV as measured from samples grown under the same conditions [95, 41]. The above figures are also consistent with an equilibrium concentration in the range $10^{12}$-$10^{13}$ cm$^{-3}$ of $V_C$ defects at 1200 °C. This is the temperature
below which the vacancy becomes immobile during sample cool down [60] and the concentration range corresponds to what is usually detected by DLTS [86, 65].

4.2. Defects in 4H-SiC produced by neutron irradiation

Characterization of defects created by ionizing radiation in epitaxial 4H-SiC layers is crucial for future improvement of radiation hardness and extending the lifetime of 4H-SiC detectors by material engineering. The change in performance of SiC-based detectors subject to irradiation by thermal and fast neutrons has been investigated in the past [29, 34, 96, 23].

The response stability of SiC SBD detectors with respect to the thermal neutron fluence was previously reported by the Westinghouse group [97, 14]. They demonstrated that the devices showed a linear response (measured as count rates) with respect to exposure to fluence rates in the range $10^1$-$10^8$ cm$^{-2}$s$^{-1}$. The relative precision of the detector (with respect to NIST-calibrated neutron fields) was about 0.6% across six orders of magnitude. In addition, the thermal-neutron response of a detector previously irradiated with a fast-neutron ($E > 1$ MeV) fluence of $1.3 \times 10^{16}$ cm$^{-2}$ was indistinguishable from that of an non-irradiated SiC detector.

More recently, Liu and co-workers [98] investigated the radiation tolerance of SiC against Si neutron detectors. The devices showed significant differences in terms of performance degradation. While Si detectors significantly degraded at a neutron fluence of $1.6 \times 10^{13}$ cm$^{-2}$ — this was quantified by a marked increase in dark current (over four orders of magnitude) and a severe (95%) reduction in the alpha-particle peak centroid, almost no degradation was found for the SiC-based detector, even for neutron fluences up to $3.8 \times 10^{15}$ cm$^{-2}$.

Performance loss of radiation detectors is usually attributed to a degradation of the life-time of carriers, which become trapped and recombine at defects. Unfortunately, the number of attempts to correlate the degradation of detection with the introduction of defects by neutron impact/reactions is rather limited [92, 34, 66]. This contrasts with the large amount of DLTS studies carried out on ion-implanted, electron- and proton-irradiated SiC samples and devices. For instance, highly energetic alpha particles (which are a common product from neutron reactions) are known to introduce
Figure 10: DLTS spectra of as-grown (pristine) and neutron-irradiated 4H-SiC SBDs with fluences of $10^{10}$ n/cm$^2$, $10^{12}$ n/cm$^2$, and $10^{13}$ n/cm$^2$ (shown as red, blue and green lines, respectively). The emission rate was 50 s$^{-1}$. Voltage settings were reverse bias $V_r = -10$ V, pulse bias $V_p = -0.1$ V and pulse width $t_p = 10$ ms. Adapted from Ref. [66].

Figure 10: DLTS spectra of as-grown (pristine) and neutron-irradiated 4H-SiC SBDs with fluences of $10^{10}$ n/cm$^2$, $10^{12}$ n/cm$^2$, and $10^{13}$ n/cm$^2$ (shown as red, blue and green lines, respectively). The emission rate was 50 s$^{-1}$. Voltage settings were reverse bias $V_r = -10$ V, pulse bias $V_p = -0.1$ V and pulse width $t_p = 10$ ms. Adapted from Ref. [66].

several defects in 4H-SiC [100].

Besides increasing the intensity of $Z_{1/2}$ and EH6/7 signals (which are related to carbon vacancies already present in as-grown material), irradiation of n-type 4H-SiC with electrons or protons leads to the appearance of a pair of DLTS peaks labeled EH1/EH3 [89, 101, 102, 103] (but also referred to as S1/S2 [90, 104] or S2/S4 [105, 106]) with activation energies of electron emission to the conduction band measured as 0.4 and 0.7 eV, respectively [100, 107]. In recent experimental and theoretical investigations, EH1 and EH3 (or S1 and S2) DLTS levels were identified as Si vacancies in different sites of the 4H-SiC crystal [108]. Interestingly, while they induce strong compensation effects, they have little impact on the charge collection efficiency of devices [105].

In our experiments, Schottky diodes were irradiated either upon bare exposure or inside Cd thermal neutron filters with a wall thickness of 1 mm. The neutron source provided fluences in the range from ($10^8$-$10^{15}$ n/cm$^2$). Figure 10 shows DLTS spectra measured on as-grown 4H-SiC (black line) and in neutron-irradiated material with
three different fluences ($10^{10}$ n/cm$^2$, $10^{12}$ n/cm$^2$ and $10^{13}$ n/cm$^2$). In as-grown material only the $Z_{1/2}$ peak is observed at around 300 K. Epithermal and fast neutron irradiation with fluence up to $10^{11}$ n/cm$^2$ did not introduce any traps detectable in the DLTS spectra within the range of temperatures considered. Peaks labeled EH1 and EH3 appear for higher neutron fluences and can be clearly observed for neutron fluences of $10^{12}$ n/cm$^2$ and $10^{13}$ n/cm$^2$.

Laplace-DLTS measurements of EH1 and EH3 did not reveal any peak splitting (due to sub-lattice location effects) although the peaks were broad. This suggests that should EH1 and EH3 arise from point defects, Laplace-DLTS was not able to resolve those located on pseudo-cubic and hexagonal sites. Despite that, activation energies for electron emission and apparent capture cross sections were determined as $\Delta E_a = 0.397$ eV and $\sigma_a = 2 \times 10^{-15}$ cm$^{-2}$ for EH1, and $\Delta E_a = 0.70$ eV and $\sigma_a = 1 \times 10^{-15}$ cm$^{-2}$ for EH3 [66]. These figures are comparable to those obtained by Alfieri and Mihaila [109], although the capture cross section of EH3 differs by about one order of magnitude. Besides the inherent large error bars in the measurement of these pre-exponential quantities, the EH3 signal overlaps the conspicuous $Z_{1/2}$ peak, making the measurement of $\sigma_a$ even more difficult.

In terms of the impact of neutron radiation on the performance of the SBD de-
tectors, temperature dependent $I$-$V$ measurements of the SBDs revealed that the fast neutron irradiation did not affect the transport properties of the detectors for neutron fluences lower that $10^{12} \text{ n/cm}^2$. This confirms the excellent radiation hardness of SiC materials. An increase in the series resistance and decrease of the capacitance of neutron irradiated samples was only observed for neutron fluences of $10^{12} \text{ n/cm}^2$ and above, followed by even more pronounced changes in the sample irradiated with a fluence of $10^{13} \text{ n/cm}^2$. The above fluence threshold correlates with the evolution of the DLTS spectrum shown in Figure 10. The increase in series resistance could therefore be related to the capture of free carriers due to the introduction of deep traps like $Z_{1/2}$ and EH1/EH3. We note that the impact of deep traps on $IV$ and $CV$ response depends on several factors like their depth within the band gap, cross sections for capture of carriers, their concentration and location within the structure. $Z_{1/2}$ is omnipresent (even in as-grown conditions) and increases in irradiated material, where its concentration is always higher than EH1 and EH3. This is probably because its production involves a lower displacement energy threshold. Although it is not clear why $Z_{1/2}$ is more harmful, a possible reason is that while EH1/EH3 are traps relatively close to the conduction band and they have acceptor character \cite{108}, the carbon vacancy ($Z_{1/2}$) also has donor levels close to mid-gap and they are expected to have considerable capture cross section for holes (minority carriers), thus leading to efficient recombination.

5. Testing of a neutron detector prototype

This section presents the experimental activities performed at the JSI TRIGA reactor (Ljubljana, Slovenia) \cite{110, 111, 112, 113}, where a set of SiC-based neutron detectors were tested, some of them being equipped with a thermal neutron converter layer enriched with $^{10}$B and $^6$LiF isotopes. The aspects regarding (1) defect production under neutron irradiation (Figure 10) and (2) detection sensitivity reported in this section were carried out in different experiments. In the study of neutron induced defects, $I$-$V$ characteristics of the irradiated diodes were measured before and after irradiation. The measured results show excellent rectifying properties before and after irradiation, which indicates that the detection properties are not expected to change appreciably.
The detectors were $4H$-SiC SBDs fabricated at the National Institute for Quantum and Radiological Science and Technology (QST, Japan). The SiC epitaxial layer was n-type doped, with thickness and nitrogen concentration in the range $25-170 \, \mu\text{m}$ and $(4.7-9.1) \times 10^{14} \, \text{cm}^{-3}$, respectively. Schottky and Ohmic contacts were fabricated by deposition and sintering of nickel front and back contacts, respectively. The SBDs had lateral dimension of $1 \, \text{mm} \times 1 \, \text{mm}$, and they were surface-mounted onto chip carriers with wire bonded electrical contacts. Figure 11 shows the components of the detector employed in the tests reported below [115]. The structures were electrically isolated by enclosure within 3D-printed plastic holders encapsulated by aluminum cases. Both the cases and holders had a window through which radiation could hit the SBDs.

The detectors were equipped with a thermal neutron converting layers consisting of $^{10}\text{B}$ and $^{6}\text{LiF}$-enriched powders. These substances contain isotopes which are among those with largest thermal neutron cross sections for $(n, \alpha)$ and $(n, t)$ reactions (around 3843 barn and 938 barn respectively, at an incident neutron energy of 0.0253 eV [116]).
Figure 13: Neutron spectra of the Pneumatic Tube irradiation channel, located in the F24 core position of the JSI TRIGA reactor. Solid red line: bare spectrum. Dashed blue line: spectrum under Cd coverage [62].

The converting materials were applied onto a plastic film and mounted above the openings of the 3D-printed SBD holders. The distance between the thermal neutron converting layers and the SBD top surface was approximately 2 mm. In the work presented in this section the thickness of the converting layers was not well controlled. This prompted us to study in detail the optimization of the converting layer thickness, and from there, to maximize the detection efficiency. This will be carried out on the basis of calculations with the SRIM (Stopping Range in Matter) and MCNP (Monte Carlo N-Particle transport) codes.

Figure 12 displays a schematic diagram of the electronic data acquisition system that was assembled for the tests. The electronic system for particle event processing and recording consisted of a preamplifier followed by shaping amplifier and a multichannel analyzer (MCA), operated by a laptop computer. In order to avoid noise from the mains power, the system was powered by a standalone battery voltage source. The latter also provided power to a separate high voltage module (HV module in Figure 12), which was used to apply a reverse bias to the SBDs.

In order to keep a low capacitance input (10 pF) to the pre-amplifier stage, we used a
CREMAT CR110-R2 pre-amplifier chip and tested the detection energy resolution with the shaping PCB module equipped with a CR-200-R2 chip with shaping times of 0.5 \( \mu \text{s} \) and 1.0 \( \mu \text{s} \). The total gain for event signal amplification was kept constant. Pole/Zero (P/Z) was adjusted for different shaping constants. Recorded events were sorted by the MCA in 2k-channel energy spectra. The performance of the detection system was observed for shaping times of 0.5 \( \mu \text{s} \) and 1.0 \( \mu \text{s} \) and reverse bias voltages of 50 V and 100 V. The combination of a reverse bias voltage of 50 V and a shaping time of 1 \( \mu \text{s} \) resulted in best spectroscopic performance. The amplitude of the reverse bias pulse (for a specific epi-layer thickness) was not optimized. That should be systematically tested in the future.

The neutron spectrum (Figure 13) was calculated by using a Monte Carlo neutron transport code MCNP [117] in conjunction with the ENDF/B-VIII.0 nuclear data library [118]. A verified and validated computational model of the JSI TRIGA reactor was employed, based on the JSI TRIGA criticality benchmark model [119], featured in the International Handbook of Evaluated Critical Safety Benchmark Experiments [120].

Neutron irradiations were performed in the Dry Chamber of the JSI TRIGA reactor. This is located in the concrete body of the reactor, being connected the core through a graphite thermalizing column [121]. It has been used for radiation hardness testing of detectors, electronic components and systems [122, 123]. In order to determine the neutron flux, activation dosimetry measurements were carried out using foils of certified reference material Al-0.1 wt.% Au, obtained from the IRMM (now Joint Research Center - JRC, Geel, Belgium). These were irradiated to measure the \(^{197}\text{Au}(n, \gamma)^{198}\text{Au} \) reaction rate, the cross-section for this nuclear reaction being a standard. The neutron flux is linearly proportional to the reactor power, which was varied between 10 kW and 250 kW (maximum steady state power). At full power the total neutron flux was \( 1.6 \times 10^7 \) n/cm\(^2\)/s. Further details may be found in Ref. 62.

Figure 14 shows the measured spectra of SiC detectors placed in the Dry Chamber of the reactor at different power levels (along with respective flux values). Spectra for detectors with either a \(^{10}\text{B} \) or \(^6\text{LiF} \) converter layers are reported. In all the recorded
Figure 14: Measured count spectra of SiC detectors placed in the Dry Chamber of the reactor at different power levels (next to respective flux values within parentheses). The thickness of the SBD epi-layer was 69 µm. (a) Spectrum of a detector covered with a $^{10}$B converter layer and (b) Spectrum of a detector covered with a $^{6}$LiF converter layer [62].

spectra a significant number of counts at higher energy channels was observed. Distinctive structures were also obtained, depending on the converter layer employed.

The relevant neutron reactions leading to the production of charged particles are either $^{10}$B(n,$\alpha$)$^{7}$Li or $^{6}$Li(n,$\alpha$)t [63]. The first one has two possibilities with respective branching ratios (BR),

$$^{10}$B + n $\rightarrow$ $\alpha$ (1776 keV) + $^{7}$Li (1013 keV); BR = 6.3%; $Q$ = 2789.5 keV
$$^{10}$B + n $\rightarrow$ $\alpha$ (1472 keV) + $^{7}$Li* (840 keV); BR = 93.7%; $Q$ = 2311.9 keV,

where $^{7}$Li$^*$ is an excited state of $^{7}$Li and $Q$ the reaction $Q$-value. Conversely, $^{6}$Li(n,$\alpha$)t follows as,

$$^{6}$Li + n $\rightarrow$ t (2731 keV) + $^{4}$He (2052 keV); $Q$ = 4783 keV.

The spectra of Figure 14 display distinctive structures, which can be assigned to the recording of secondary charged particles originating from $^{10}$B(n,$\alpha$) and $^{6}$Li(n,t) reactions. The main features (regions) in the spectra are denoted as “R0-4” and “R0-2” in Figures 14(a) and 14(b), respectively. The sharp peak R0 that is present in both
spectra at low energy channels is attributed to electronic noise. Inspection of the kinetic energy of the reaction products against the spectra allowed a tentative assignment of the spectral features.

For the $^{10}$B-covered detectors [Figure 14(a)], R1 was connected to hits by $^7$Li ($E = 1013$ keV) and $^7$Li$^+$ ($E = 840$ keV) ions, R2 was assigned to alpha particles ($E = 1776$ keV or $E = 1472$ keV), R3 was assigned to a combined detection of alphas and $^7$Li$^+$ from the dominant reaction, while R4 was attributed to the analogous combined detection of alphas and $^7$Li from the less probable reaction branch.

The above assignments are tentative, as there is observable overlap between regions and no clear peaks appearing. This is probably due to (i) a partial energy loss of the secondary particles in the converter / air gap / front contact, and (ii) a limited detection resolution. The assignments are made on the basis of the drops in the pulse height spectra observed on the high energy side of each region. R1 drops off at slightly above 1000 keV, corresponding to the maximum $^7$Li energy (1013 keV), R2 drops off at approximately 1700 keV, corresponding to the maximum energy threshold of alpha particles (1776 keV). R3 in particular drops off very sharply at approximately 2300 keV, corresponding very well to the maximum detectable particle energy from the dominant $^{10}$B(n,$\alpha$) reaction channel (1472 keV + 840 keV = 2312 keV). R4 has the least number of counts, and seems to end at above 2700 keV, corresponding to the maximal detectable particle energy from the less probable reaction channel (1776 keV + 1013 keV = 2789 keV).

For the spectra obtained using the $^6$LiF converter layer [Figure 14(b)], the features were not so well resolved and the interpretation was more difficult and tentative. Accordingly, the R1 plateau was connected to partial energy deposition events, while R2 was attributed to a combination of partial energy deposition from tritons ($E = 2731$ keV) and alpha particles ($E = 2052$ keV).

Among the most important specifications of a neutron detector are its sensitivity and response linearity. In order to minimize electronic noise of a real-life detection system, the signal recorded should be derived from the total counts above a certain channel number (energy threshold). Radulović and co-workers [62] set this threshold at around 600 keV, which is definitely above the R0 peak of Figures 14(a) and 14(b),
and obtained total detected count rates as a function of reactor power and corresponding neutron flux as depicted in Figure 15. The figure shows excellent response linearity, irrespectively of the converter layer employed or the thickness of the epitaxial $4H$-SiC front layer. The sensitivity of the detector per unit of neutron flux is given by the slope in the graphs. On average, these were found to be over $10^{-5}$ (counts/s)/(n/cm$^2$s) [62]. In Figure 15, the data labeled with “170 µm, $^{10}$B (1)” and “170 µm / $^{10}$B (2)” refer to separate measurements with the same 170 µm thick detector, but different $^{10}$B converters. The difference in the results suggests a dependence of the flux of ionizing particles hitting the SiC diode with respect to the converter layer thickness.

It becomes apparent from Figure 15 that for the same $^{10}$B conversion layer, the thinner epi-SiC layer leads to higher sensitivity. We note that the sensitivity for thermal neutrons is mostly determined by the properties of the conversion layer. Hence, given (i) the limited amount of samples and (ii) the lack of detail regarding the characterization of the conversion layers, we cannot draw a conclusion regarding thickness-sensitivity trends. In the present work, the detection sensitivity is primarily dependent on the use of either $^6$LiF or $^{10}$B layers. The decay products of the $^6$Li include highly energetic tritons, whose penetration depth and efficiency in terms of electron-hole creation could be more favorable than that of the alpha particles from $^{10}$B.

Improvements to the sensitivity of detectors based on SiC-SBD could in principle be achieved by increasing the cross-section area of the active region, i.e. by enlarging the area of the semiconductor layers. This route is hindered by two factors, namely (i) a decrease in the fabrication yield and (ii) an increase of the SBD capacitance which at some point becomes detrimental and lowers the sensitivity. Future large-area semiconductor-based detectors are therefore likely to be pixelized. However, this has the disadvantage of increasing the complexity of fabrication and maintenance, including a much more complex read-out electronics and data processing.

The neutron fluxes considered in the measurements of Ref. [62] are at least 6 orders of magnitude larger than the background neutron flux at the Earth surface [124]. This is the critical benchmark which any detector relevant for home-land security will have to comply with, particularly if the aim is to screen for disguised radiological materials. Commercially available $^{10}$BF$_3$ and $^3$He detectors show sensitivity values of the order
of 4 (counts/s)/(n/cm²s) and 10-100 (counts/s)/(n/cm²s), respectively. Their size are in range 2.5-5.1 cm (1-2 inches) in diameter and 0.3-1.8 m long. For comparison, a 100 × 1000 pixelized SiC detectors with the above reported sensitivity, would show an overall sensitivity of around 1 (counts/s)/(n/cm²s). We must mind though, that current neutron sensitivity of SiC-based detectors is limited and its application on the field would imply very large detector arrays, which in turn would require a high degree of system complexity and serious technical challenges related to the implementation and operation.

6. Conclusions

We presented a review about the workings of SiC-based neutron detectors along with several issues related to material properties, device fabrication and testing. The paper summarizes the work carried within the E-SiCure project (Engineering Silicon Carbide for Border and Port Security), funded by the NATO Science for Peace and Security Programme. The main goal was the development of material and device technologies to support the fabrication of radiation-hard silicon carbide (SiC) based detectors of special nuclear materials, envisaging the enhancement of border security and customs screening capability. Achievements include the fabrication of a 4H-SiC based SBD detector equipped with a thermal neutron converter layer, as well as the characterization of the main carrier traps affecting the performance before and after exposure of the devices to neutron fields.

We started the first section of the paper by justifying the significance of developing a neutron detection technology that could match that based on ³He. The advantages of semiconductor-detectors were enumerated, in particular those of devices based on the 4H polytype of silicon carbide (4H-SiC), including its radiation hardness and electronic specifications. We described the basic structure of a detector based on a 4H-SiC Schottky barrier diode covered with a thermal neutron converter layer. Here we went through the main physical processes taking place, from the impact of a neutron until the collection of charge carriers at the terminals. The importance of understanding and controlling the presence of defects in the semiconductor was highlighted based on their
impact on the carrier life-time and sensitivity of the detector.

In Section 2 we revised the main properties of SiC materials, namely its polytypism and introduced the common crystallographic notations employed. The optical and electronic transport properties of SiC were also described, including the band gap width and its dependence with the polytype. We then proceeded to Section 3 where the material (4H-SiC) growth and device fabrication was addressed. We started by describing the different types of growth techniques involving seeding as well as bulk and epitaxial growth. The fabrication of a Schottky barrier diode was described, namely the formation of Schottky and Ohmic contacts.

We dedicated considerable effort to the identification, characterization and modeling of defects in as-grown and neutron irradiated 4H-SiC. The defect which is most detrimental to the electronic performance of the devices gives rise to a prominent signal detected by deep level transient spectroscopy. This signal is commonly labeled as \( Z_{1/2} \), and arises from a carbon vacancy. A total of four electronic transitions in the gap were resolved experimentally. With the help of theoretical modeling, they were all assigned to carbon vacancies in different lattice sites and charge states. Controlling the concentration of this defect seems to be critical to improve the performance of the diodes.

The development and testing of a SiC SBD neutron detector prototype and acquisition system was described in Section 5. It was shown that detectors based on SiC SBDs and equipped with thermal neutron converter films (enriched with \(^{10}\)B and \(^{6}\)LiF isotopes) clearly show a neutron response with excellent linearity. Arrays of such detectors are anticipated to offer sensitivities close to those currently available in the market.

The above results stand as a motivation for future improvements of SiC-based neutron detectors. In fact there is ample room for improvement and several key factors influencing the sensitivity of the detectors are worthy of investigating. These include decreasing the concentration of deep carrier traps during growth, optimizing the charge collection active volume, avoiding an air layer between the converter material and the SBD top surface by enclosing the device in a vacuum chamber, optimizing the thick-
Figure 15: Total detected count rates above channel no. 500 (around 600 keV), as a function of reactor power and respective neutron flux. Four different diodes with varying thickness of the 4H-SiC layer and type of converter materials (see legend) were considered. Adapted from Ref. 62.

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