

Point defects in silicon carbide

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A review is given on recent progress made in a microscopic understanding of point defects in silicon carbide (SiC). Defect structures to be discussed include shallow nitrogen donors, group-III acceptors, the transition metal impurities vanadium and titanium and radiation induced defects, like the silicon vacancy in SiC.

1. Introduction

We are presently observing a reinspection of silicon carbide (SiC) as a promising material for semiconductor devices operating under extreme conditions. This development obviously demands enforced materials research on this fascinating semiconductor. In particular, the role of electrically active shallow and deep point defects in SiC is, in many aspects, still poorly understood.

It is the purpose of this paper to review and highlight some recent progress made in the microscopic understanding of point defects in silicon carbide. Defect structures to be discussed are shallow donor and acceptor dopants, transition metal impurities and intrinsic lattice defects, which can also be created under fast-particle irradiation.

Emphasis will be laid on results obtained by experimental techniques which enable microscopic identification of the defects structure, i.e. photoluminescence spectroscopy and, in particular, magnetic resonance techniques, as electron spin resonance (ESR) and optically detected magnetic resonance (ODMR).

2. Nitrogen donors

Nitrogen is the standard n-type dopant for

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SiC. The nitrogen donors are assumed to substitute for carbon in the SiC-lattice. The ionisation energies E_D of N-donors on the various C-sites in a given SiC-polytype may vary considerably. Recent infrared spectroscopic studies [1] locate the nitrogen donors ground state in 6H-SiC at $E_D = E_C - 81.0$ meV, 137.6 meV and 142.4 meV for the hexagonal (h) and quasi-cubic (k_1, k_2) lattice sites, respectively. The energetical position of the excited, effective mass like donor states $2p_{\pm}$, $2p_0$, $3p_{\pm}$, $3p_0$ could also be inferred from the infrared spectra [1].

Because of the multi-valley structure of the conduction band of SiC, a valley-orbit splitting Δ_{VO} of the donors 1s ground state occurs. This splitting has been determined, already some time ago [2], by electronic Raman scattering, for n-type 6H-SiC. Surprisingly, it was found that Δ_{VO} for the hexagonal site, 13.0 meV, is much smaller than for the two quasi-cubic sites, 60.3 meV and 62.6 meV [2]. The situation is illustrated in fig. 1.

Early ESR-investigations [3] of the nitrogen donors in 6H-SiC revealed practically only one type of ^{14}N -hyperfine triplet, in spite of the threefold site multiplicity in 6H-SiC. From this it was concluded that the spin density of the donor electron at the three ^{14}N nuclear sites h , k_1 , k_2 is almost equal. However, more recent ESR-investigations [4], also performed at higher microwave frequencies (142 GHz) [5], have shown that such conclusion is wrong. Instead, it was established [4] that the spin density, as reflected by the ^{14}N -hyperfine splitting, at the hexagonal (h) site

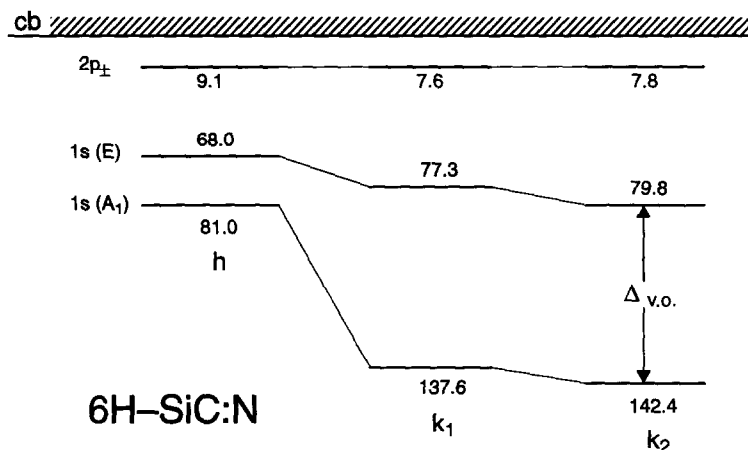


Fig. 1. Energy levels (meV) of nitrogen donors in 6H-SiC on the hexagonal (h) and quasi-cubic (k_1, k_2) lattice sites. The valley-orbit splitting $\Delta_{v.o.}$ was determined by electronic Raman scattering [2], the position of excited effective-mass-like states, like $2p_{\pm}$, by infrared absorption [1].

is only about one tenth of that occurring at the two quasi-cubic (k_1, k_2) lattice sites in 6H-SiC, see fig. 2. Similar observations have been made for nitrogen donors in 4H-SiC and 15R-SiC [4]. A theoretical interpretation of these surprising fluctuations has still to be given.

In cubic 3C-SiC, where only one type of lattice site exists, the ionisation energy of the nitrogen donor is rather low, $E_D = E_C - 53$ meV. It should be noted that in nitrogen-doped 3C-SiC epitaxial layers the presence of an even more shallow donor species has been repeatedly reported [6].

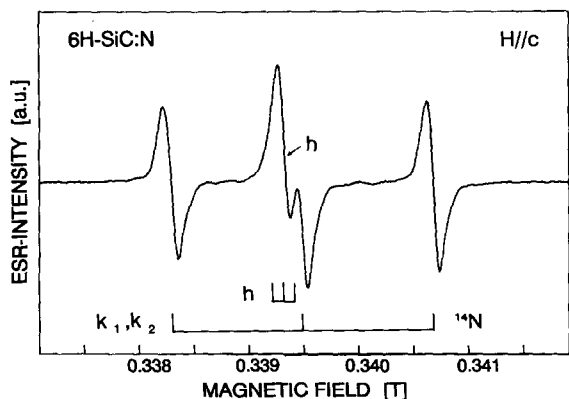


Fig. 2. ESR spectrum (9.5 GHz) of nitrogen donors in n-type 6H-SiC:N on the quasi-cubic (k_1, k_2) and hexagonal (h) lattice sites, at 70 K. The much smaller ^{14}N hyperfine splitting of line h is indicated; it can be resolved at lower temperatures [4].

Its microscopic structure remains to be elucidated.

By ESR it was recently detected that nitrogen donors in 6H- and 4H-SiC show a strong preference for association with titanium trace impurities [7,8,9], common in Lely-grown SiC crystals. The resulting ($\text{N}_C\text{-Ti}_{\text{Si}}$) nearest neighbor pair acts as deep donor. Its electrical activity arises from the excess 3d-electron in the $\text{Ti}(3d^1)$ configuration, which is created after the nitrogen donor electron has collapsed from its delocalized 1s orbit into the localized 3d orbital of the titanium ligand. Photo-ESR experiments [8,9] performed on a Ti-N pair in 6H-SiC indicate that the resulting deep donor level is located at $E_C - 0.6$ eV. This value greatly exceeds those of isolated shallow nitrogen donors in 6H-SiC, which are in the 0.08 eV–0.14 eV range, as quoted above.

3. Group-III acceptors

Aluminum, substituting for silicon, is the preferred p-type dopant for SiC bulk crystals and epitaxial layers. The ionisation energies of the Al-acceptors have been determined by donor (N)-acceptor (Al) photoluminescence [10]; they were found to lie in the 0.25 eV range, depending on lattice site (h, k) and polytype. The ac-

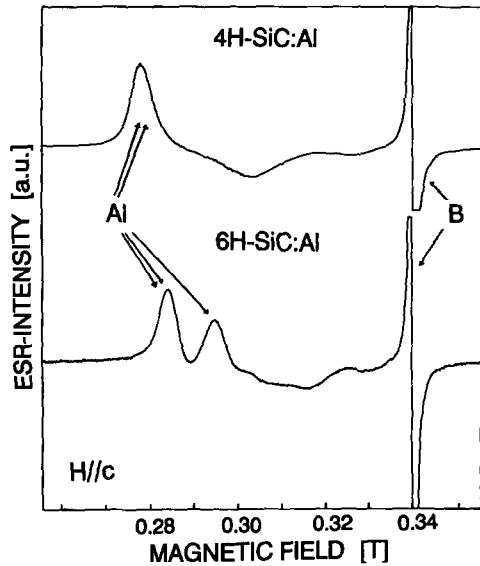


Fig. 3. ESR spectra (9.5 GHz) of aluminum acceptors in p-type 4H-SiC:Al and 6H-SiC:Al, at 5 K. The assignment of the individual Al-signals to specific lattice sites is not unique. Note the strong ESR signal arising from inadvertent boron (B) acceptor contaminations [13].

ceptor states of *gallium* in SiC have been found [10] to be about 0.1 eV deeper than those formed by the aluminum acceptors.

Apart from the photoluminescence, Al and Ga acceptors, in the noncubic modifications of SiC, can also be assessed by magnetic resonance techniques, as ESR and ODMR. This is remarkable in a sense, since ESR or ODMR of neutral acceptors in cubic semiconductors, as Si and GaP, can, in general, only be observed after uniaxial stress is applied to the crystal. In this way, the fourfold Γ_8 degeneracy of the acceptor ground state, which is very sensitive to random crystalline strains, is removed. Of course, in the noncubic modifications of SiC this degeneracy is already lifted and magnetic resonance of neutral acceptors can therefore be readily observed.

Al acceptors have been detected by ODMR [11,12] in 6H-SiC, and also by conventional ESR [13], see fig. 3. The acceptor g factor was found to follow a law of the type $g = g_{\parallel} \cos \vartheta$, where ϑ is the angle between the static magnetic field and the crystalline c axis. Such behaviour is expected for effective-mass-like acceptors in 6H-SiC and

other noncubic polytypes, where the delocalized hole moves in the upper Γ_8 valence band [11]. Similar ODMR-observations have been made [12] for gallium acceptors in 6H-SiC.

Scandium forms rather deep acceptor states in silicon carbide, at $E_v + 0.55$ eV in 6H-SiC [14]. Sc doping has therefore been occasionally used for producing yellow-emitting material. The atomic configuration of Sc is $3d^1 4s^2$, whereas that of Al and Ga is $3s^2 3p^1$ and $4s^2 4p^1$, respectively. This may account for the different electronic structure of the Sc acceptor, which is also manifested by a rather small anisotropy of its electronic g factor, as determined by ODMR [12].

The case of *boron* acceptors in SiC deserves special attention. First of all, it should be noted that boron is a practically unavoidable trace contamination in most Lely-grown SiC crystals, as recently [13] evidenced by ESR, see fig. 3.

The ESR-spectrum of boron acceptors in 6H-SiC, first reported [3] in 1961, has been repeatedly reinterpreted in the past. It was originally [3] assigned to carbon substitutional boron acceptors, having point symmetry C_{3v} on all (h, k_1, k_2) lattice sites. However, subsequent ESR studies [15,16] have revealed that the boron centers undergo a Jahn–Teller type displacement along the ligands bond direction. In this way, their point symmetry can be lower than C_{3v} . Furthermore, by an ESR-analysis of ^{13}C -enriched SiC polytypes, it became apparent that the ESR-active boron centers substitute for silicon [16] and not for carbon, as previously assumed [3].

The g factors of the ESR-active boron centers in SiC-polytypes are very close to the free-spin value, 2.0023. This may be taken as evidence that one is dealing with a deep level defect. In contrast, the g factors of the more shallow Al and Ga acceptors in 6H-SiC are highly anisotropic, as mentioned above.

The question of the position of boron-related acceptor levels in the bandgap of SiC polytypes is presently still under debate. In 6H-SiC:B a deep level at $E_v + 0.6$ eV and also a more shallow level at $E_v + 0.3$ eV have been observed by deep level transient spectroscopy (DLTS) [17]. A definitive assignment of these levels to a

specific, boron-related defect structure is still lacking.

4. Transition metals

Transition metals are omnipresent and practically unavoidable trace impurities in most semiconductors. Because of the instability and multiplicity of their charge state, they can act as electrically active deep level defects. It is therefore not surprising that extensive research work during the last decades has led to an almost encyclopedic knowledge on the behaviour of transition metal point defects in silicon.

However, in silicon carbide such understanding is still in its infancy. For example very little is known about the presumed electrical activity of transition metal impurities in SiC. This lack of knowledge is surprising, and challenging, in view of the fact that transition metals, in particular *titanium* and *vanadium* are practically unavoidable trace impurities (in the 10 ppm range!) in Lely-grown SiC-crystals [18]. The origin of such contaminations during crystal growth is presently still under debate. Obviously, identification and control of transition metal deep-level impurities is prerequisite for satisfactory performance of electronic and opto-electronic devices fabricated from silicon carbide.

We will now shortly review some salient features of vanadium and titanium point defects in hexagonal SiC polytypes, as studied by electron spin resonance (ESR) techniques and by optical spectroscopy. A more detailed discussion has been given before [9].

4.1. Amphoteric vanadium

By ESR we have recently detected [8,9,19] that omnipresent vanadium contaminations in Lely-grown SiC crystals act as amphoteric deep level impurities, substituting the various silicon sites in the lattice. Three charge states of vanadium were found to exist in SiC: $V^{3+}(3d^2)$, $V^{4+}(3d^1)$ and $V^{5+}(3d^0)$, their relative occurrence depending on the position of the Fermi level. In n-type 4H- and 6H-SiC, the ESR-spec-

trum of the ionized acceptor (A^-) charge state $V^{3+}(3d^2)$, $S=1$ is dominant. No vanadium-related ESR signals are detectable in p-type SiC, indicating the occurrence of vanadium in its pentavalent diamagnetic charge state $V^{5+}(3d^0)$, which corresponds to that of an ionized donor (D^+). Thus vanadium in SiC acts as an electrically amphoteric impurity, introducing two levels, $D^0/D^+ \triangleq V^{4+}/V^{5+}$ and $A^-/A^0 \triangleq V^{4+}/V^{3+}$ in the bandgap. The role of vanadium as a minority-carrier lifetime killer in SiC-based optoelectronic devices has been suggested from these results.

The position of the donor level $V^{4+}/V^{5+} \triangleq D^0/D^+$ in the bandgap of 6H-SiC has been determined by photo-ESR experiments. For vanadium occupying the quasi-cubic sites k_1 and k_2 in 6H-SiC a value close to midgap, $E_V + 1.6$ eV, has been obtained [8,9]. A similar value, $E_V + 1.5$ eV, was obtained by infrared photo-neutralisation experiments [20] performed on the same crystal as used before in the photo-ESR study. In this way, also the position of the vanadium donor level for the hexagonal site in 6H-SiC could be located at $E_V + 1.3$ eV [20]. The position of the vanadium acceptor level $V^{3+}(3d^2)/V^{4+}(3d^1) \triangleq A^-/A^0$ could so far not be determined. We presume it to be located in the upper third of the bandgap.

We have discovered [19] that *all* Lely-grown SiC crystals investigated so far by us exhibit a characteristic infrared luminescence in the 1.3–1.5 μm spectral range. Typical spectra [21] for 4H-, 6H- and 15R-SiC polytypes are shown in fig. 4. This infrared luminescence arises from intra 3d-shell transitions, ${}^2T_2 \rightarrow {}^2E$, of neutral, silicon-substitutional vanadium, $V^{4+}_{\text{Si}}(3d^1)$. The multiplicity of zero-phonon lines, as apparent in fig. 4 and also resolved already at 77 K, reflects immediately the site multiplicity characteristic for an individual SiC polytype. This feature can be exploited for quick, and spatially resolved, morphology determination of SiC polytype crystals [21].

It is interesting to note that the intra 3d-shell luminescence of neutral vanadium is also observed in n-type or p-type material, where the neutral charge state does not exist. In these

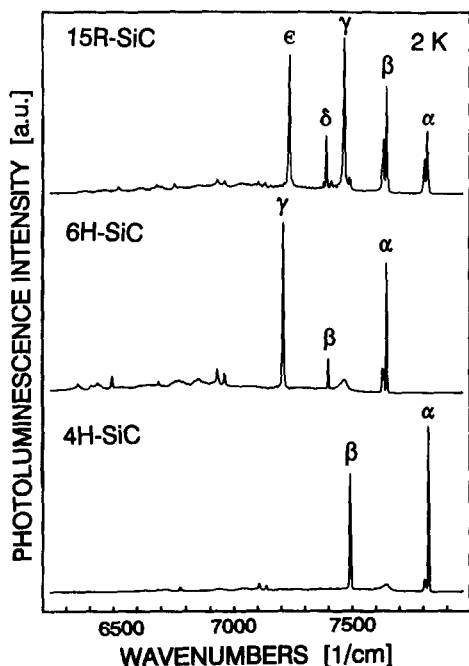


Fig. 4. Near-infrared photoluminescence spectra of vanadium trace impurities in 4H-, 6H- and 15R-polytype SiC crystals, at 2 K. The zero-phonon lines arise from intra-3d-shell transitions, ${}^2T_2 \rightarrow {}^2E$, of $V^{4+}_{Si}(3d^1)$ occupying the substitutional silicon sites in the SiC lattice. Intrinsic and vanadium-induced phonon sidebands are observed at lower photon energies [19,21].

cases, photo-neutralisation of the $A^-(3d^2)$ - and $D^+(3d^0)$ -states of vanadium must be enforced by the exciting light, before luminescence of the neutral $3d^1$ -state can occur. In contrast, the complementary infrared absorption, ${}^2E \rightarrow {}^2T_2$, of neutral $V^{4+}_{Si}(3d^1)$ has been observed only in high-resistivity or weakly p-type crystals [19,20].

We finally mention that the characteristic intra 3d-shell infrared luminescence of $V^{4+}_{Si}(3d^1)$, inevitable in hexagonal and rhombohedral SiC polytypes, could not be observed in cubic 3C-SiC epitaxial layers. A likely explanation for this failure may be a resonant position of the luminescent 2T_2 -level in the conduction band of 3C-SiC, $E_g = 2.4$ eV.

4.2. Titanium

The identification of titanium as luminescent

center responsible for the intense green below bandgap emission of silicon carbide (SiC) crystals, see fig. 5, has an interesting and controversial history. In the model of Patrick and Choyke [22] it is now agreed that silicon-substitutional isoelectronic neutral $Ti(3d^0)$ impurities bind excitons. Before recombination, the electron is trapped in the $3d_e$ -orbital, thus forming the localized A^- -state $Ti(3d^1)$. In contrast, the hole wavefunction is more delocalized, being bound to the A^- -core only by Coulombic attraction. More detailed studies [23] by optically detected magnetic resonance (ODMR) have further revealed that the luminescent state of the green emission in 4H-SiC and 6H-SiC is a spin-triplet, $S = 1$, whereas the ground state is diamagnetic, $S = 0$. However, up to now no direct experimental evidence was given that the presumed ionized acceptor state of titanium, $Ti^{3+}(3d^1)$, $S = 1/2$, really lies in the bandgap of silicon carbide.

Recent ESR-work has revealed that the A^- -

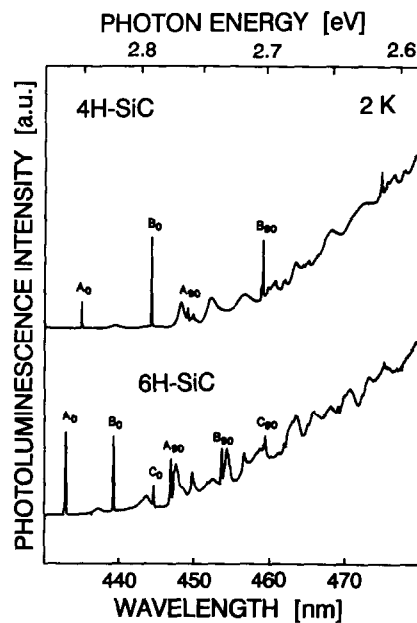


Fig. 5. Photoluminescence spectra of titanium trace impurities in 4H- and 6H-polytype SiC crystals. The zero-phonon lines A_0 , B_0 and C_0 arise from exciton recombination at $Ti^{4+}_{Si}(3d^0)$, occupying the substitutional silicon sites in the SiC lattice. Lines labelled A_{90} , B_{90} and C_{90} correspond to local phonon (~ 90 meV) sidebands.

state $\text{Ti}^{3+}(3d^1)$ is stable only in 4H-SiC, $E_{gx} = 3.265$ eV, but not in 6H-SiC, $E_{gx} = 3.023$ eV, and other lower bandgap SiC polytypes [9]. The deep acceptor level $(-1/0) \triangleq \text{Ti}^{3+}(3d^1)/\text{Ti}^{4+}(3d^0)$ is estimated [9] to be located only a few tenths of an eV below the conduction band edge of 4H-SiC.

We have already mentioned in section 2 that titanium impurities in 4H- and 6H-SiC show a strong tendency to complex with nitrogen donors. The resulting $\text{Ti}_{\text{Si}}-\text{N}_{\text{C}}$ nearest neighbor pair in 6H-SiC has been found, by photo-ESR, to act as deep donor [8,9]; its occurrence and electrical activity in other SiC polytypes remains to be investigated.

5. Radiation-induced defects

The primary defects created by fast-particle irradiation of solids are vacancies and interstitials. In a binary compound, as SiC, also the third kind of elemental lattice disorder, i.e. anti-site defects, has to be considered.

In *silicon*, primary radiation defects are mobile at temperatures far below 300 K. Consequently, the defect scenery observed after room temperature irradiation is mainly represented by associated radiation defect centers, as divacancies or vacancy-donor complexes. Most of these radiation-induced defects in silicon are thermally annihilated at temperatures above 300°C.

In *silicon carbide* the situation is markedly different from that encountered in silicon. Because of the much higher activation energy for interstitial and vacancy diffusion in this binary and 'hard' compound, primary radiation defects, in particular isolated vacancies, are thermally stable at room temperature, and far above. Thus, it has been recently established [24,25] by ESR that the silicon vacancy in 3C-SiC is thermally stable up to 750°C. Under annealing at higher temperatures the mono-vacancies become mobile and can form associated intrinsic or extrinsic point defect pairs, as divacancies or vacancy-impurity complexes. We anticipate that some of these secondary deep-level complexes may act as very efficient luminescent centers.

5.1. The silicon vacancy in SiC

After fast-particle irradiation at room temperature, a characteristic ESR-signal at $g = 2.003$ had been observed [26] in 3C-SiC and 6H-SiC, see fig. 6. The microscopic structure of this ESR-active radiation defect remained debated over decades. Only recently, after detection of a weak ^{13}C -ligand hyperfine structure in the ESR-spectrum [24], it became apparent, that the defect responsible is the isolated silicon vacancy, V_{Si} . The majority of radiation-induced V_{Si} defects in 3C-SiC was found to anneal at 750°C, in a first-order reaction with an activation energy of 2.2 eV [25]. This value greatly exceeds those typical for isolated-vacancy-center motion in silicon [27]. Some partial annealing of the V_{Si} defect in SiC was observed already at 150°C, presumably due to recapture of interstitials by V_{Si} .

A dilemma remains. The assignment of the isotropic ESR-spectrum described above to an isolated, undistorted (T_d) silicon vacancy in SiC, having electronic spin $S = 1/2$, is in conflict with experimental observations on isolated vacancy centers in silicon and diamond. In silicon, the electronic ground state derives from a t_2 -state, $S = 1/2$, subject to further symmetry reducing Jahn-Teller distortions [27]. In contrast, very recent ESR and ENDOR work [28] on the V_{C}

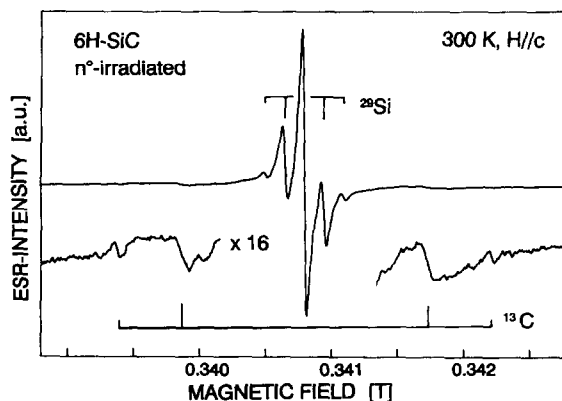


Fig. 6. ESR spectrum (9.5 GHz) of the isolated silicon vacancy V_{Si} in fast-neutron irradiated 6H-SiC, at 300 K. Hyperfine satellites arising from four nearest ^{13}C and twelve next-nearest ^{29}Si ligands are apparent.

carbon vacancy in diamond revealed that its electronic ground state is the high spin state 4A_2 , $S = 3/2$, which demonstrates the importance of exchange interaction for this vacancy.

In view of such experience on vacancy centers in silicon and diamond, the existence of low-spin ($S = 1/2$) nondistorted (T_d) silicon vacancy centers in 3C-SiC and hexagonal polytypes remains a puzzle. Possibly, a dynamic Jahn–Teller effect occurs in the t_2 -derived ground state of V_{Si} which may result in an ESR-spectrum having full T_d symmetry.

5.2. Vacancy interactions with other defects

At temperatures above 750°C, when the silicon vacancy in SiC becomes mobile, complexing of V_{Si} with other intrinsic point defects is likely to occur. As in the case of secondary radiation defect centers in silicon [27], complexes of the type V_{Si} –oxygen, V_{Si} –donor, V_{Si} –acceptor, as well as divacancy centers, may be formed. However, a microscopic understanding of such defect complexes in SiC does so far practically not exist.

Remarkably, one type of the secondary radiation complexes in SiC has been found to be strongly luminescent (D_1 -center) [29]. The maxi-

mum photon energy of its characteristic emission band depends directly on the bandgap energy of the SiC-polytype [30], see fig. 7. The D_1 -center has also been detected as *native* defect in as-grown 3C-SiC epitaxial layers deposited on silicon substrates [31]. In spite of a detailed magneto-optical study [32] of the D_1 zero-phonon lines in 6H-SiC, a definite microscopic model for the luminescent center in question does not yet exist. Apart from divacancy-related defects [29,32], also V_{Si} – N_C vacancy–donor complexes [33] have been suggested.

We should finally mention that hydrogen (or deuterium) implantation in SiC, followed by high-temperature annealing, can lead to another type of efficient near-bandgap emission [29]. The luminescent center responsible has been identified [29] as a silicon vacancy, in which a hydrogen (or deuterium) atom is bound to one of the four carbon ligands around V_{Si} .

6. Antisite defects

Antisite disorder is expected to be typical for as-grown SiC-crystals since, in contrast to III–V compounds, both lattice atoms belong to the same group of the Periodic Table. Indeed, recent theoretical studies [34] predict rather low (~ 3 eV) energies of formation for both types of antisite defects Si_C and C_{Si} in 3C-SiC. Thus, non-stoichiometry in SiC may be preferentially realized by antisite formation. Alternatively, antisite defects should also be easily formed under fast-particle irradiation of SiC.

Another point is the question of a possible electrical activity of antisite defects in SiC. In III–V compounds as GaAs, the electrical activity of the deep donor $As_{Ga}/EL2$ anion–antisite defect is now known to be of paramount importance. Similar experience on antisite defects in SiC does not exist at the present time. However, theoretical studies [34,35] indicate that both types of *isolated* antisite defects, Si_C and C_{Si} , in 3C-SiC are electrically inactive. Of course, further experimental work is compulsory to clarify this important question.

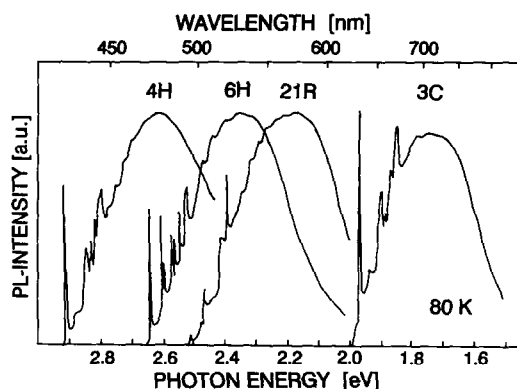


Fig. 7. Photoluminescence (D_1) bands emitted by various SiC polytypes after fast-neutron irradiation and subsequent thermal annealing at 1200°C [30]. The luminescent center responsible is a secondary radiation defect whose microscopic structure could not yet be definitively established.

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