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Study of indium–defect interactions in diamond using two-dimensional conversion-electron emission channelling

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Abstract. Channelling has, since its inception, proven to be a valuable tool in locating the geometric position of atoms in the crystal lattice. Allied with powerful theoretical models, it can yield detailed information on the positions that these impurities occupy. ^{111}In, a radioactive isotope with a conveniently short half-life, is an often-used probe of heavy-atom doping of materials. Previous work has centred on the lattice location of ^{111}In implanted in type IIa diamond. Theoretical calculations on this 'pure' system have also recently been made. We have performed the first studies of ^{111}In implanted into various carefully selected, defect-rich diamond systems and obtained fractions for the sites occupied. The defect systems investigated include nitrogen in various configurations, boron, hydrogen and vacancies. The use of two-dimensional conversion-electron emission channelling (CEEC) has enabled the system to be studied in greater detail than with conventional one-dimensional CEEC. Coupled with the acquisition of the CEEC spectra for all the major channelling axes, this has yielded a comprehensive data set. The spectra are consistent with a pure substitutional fraction as well as another fraction, approximately 0.45 Å from the substitutional along a $\langle 111 \rangle$ direction. Previous measurements observed these two components together as substitutional or 'near-substitutional'. The data have been compared to simulated CEEC spectra and earlier quantum chemical calculations. The pure substitutional fraction is indicated to be in a defect-free configuration while the component displaced away from substitutional involves most probably the divacancy and another nearby defect. The results show no dependence on impurity type, even after annealing. If indium complexation with these defects does occur it is shown not to measurably affect the channelled spectra and thus the projected lattice location of the ^{111}In probe. The origin of the random fraction measured in previous studies is proposed to be partially due to In in different multi-vacancy complexes. Taken together, the data indicate the importance of vacancies (complexes) in the final configuration for In after implantation in diamond.

1. Introduction

For any (semiconductor) material, knowledge of the behaviour of impurities, in particular of dopant–impurity complexes, is of importance. The exact conditions of implantation and subsequent annealing have been shown to be of importance in ion implantation in diamond [1, 2]. In order to achieve n-type doping of diamond, some attention is switching to heavier

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dopant atoms [3], in the belief that they might generate shallow dopant states which would be suited to the manufacture of diamond electronic devices. Most recently the location of ^{33}P has been the focus of conversion-electron emission channelling (CEEC) measurements [4, 5]. A fraction of 70 $\pm$ 10% of substitutional P was obtained, which is in agreement with results showing P as an n-type dopant [6].

The use of channelling to determine the lattice sites of elements of interest in crystalline materials is well known. In particular emission channelling has a number of distinct advantages over the more usual ion beam channelling [7]. Amongst others only a very low concentration of probe atoms (i.e. impurities) is required, less than 10^{13} atoms cm^{-2}. In diamond this corresponds to a concentration at the implantation peak of about 50 ppm (atomic), at the implantation energies commonly used ($\sim$60 keV). The number of emitted particles needed for analysis is thus orders of magnitude less than that in an analysing beam, vastly reducing radiation damage. In the case of electrons with energies below a few hundred keV, due to their low momenta, radiation damage is avoided during analysis. The low damage due to the radioactive impurity implantation may then be mostly annealed out before the analysis begins. This allows also the study of the effects of annealing on radiation damage.

^{111}In has been a common probe for studying the interaction of heavy ions in the diamond lattice [8–11]. A two-threshold annealing cycle has been identified in the case of diamond for the In site [8, 9, 11], with the first threshold occurring at between 300 and 600 K and the second after 1200 K. These results indicated a large (between 35 and 60%), substitutional or very near-substitutional (less than approximately 0.1 Å away), fraction. The remainder of the In atoms occupied random or irregular sites. The nature of these sites was not clear. The possibility of a significant tetrahedral interstitial site has been ruled out by earlier emission channelling results [11]. Theoretical local density pseudopotential calculations have shown the most stable site to be an interstitial bond-centred position between two adjacent carbon vacancies [12]. The presence of this divacancy stabilized this system. The obvious configurations without a vacancy (substitutional and bond-centred) were found to be unstable or metastable. The importance of vacancies in the doping of diamond has been previously documented [1, 2]. Theoretical studies have indicated that these might impose a limit on the ability to dope diamond with heavy n-type ions [13]. Studies on In in germanium have shown the formation of both In–vacancy and In–self-interstitial complexes [14–16], with acceptor and donor properties respectively being suggested [16]. The existence of different charge states of the In–vacancy complex in germanium is also proposed [15]. The authors of these papers have all suggested that single vacancies were involved, although no reasons were given for two or more vacancies not being constituents of the complex.

For silicon a particular relationship was found to exist between group-III and group-V dopants [17]. If a group-III dopant was implanted into a crystal that already had a group-V dopant then the group-III atoms occupied a much greater fraction of substitutional sites than if they were implanted alone into the pure crystal, and in addition no interstitial sites were discernible. This has been explained in terms of the formation of local chemical bonds between the acceptor and the donor atoms [18]. The charge states of the Frenkel defects resulting from the implantation are of importance in determining the Fermi level and the resultant defect reaction kinetics. Perturbed angular correlation measurements on silicon doped with In and As showed the formation of In–As pairs [19]. It was hypothesized that the formation of these pairs pulled the In atoms onto substitutional sites. We have implanted In atoms into diamond containing another group-V element, namely nitrogen. In this case the sample was a type IaAB diamond, containing naturally occurring nitrogen in the form of A and B centres. An A centre is argued to be composed of two nearest-neighbour substitutional nitrogen atoms [20], whereas a B centre is thought to be four substitutional nitrogen atoms surrounding a vacancy

[21, 22]. A centres have been speculated to be deep-lying donors in diamond [23]. We have also implanted In into type IIb diamond which naturally has a group-III dopant already in place, namely boron, to see if any competition for substitutional sites or any other defect–defect interaction occurs. This also serves to investigate whether the p-type semiconducting nature of this sample, and thus the resultant differing Fermi level, influences the In lattice site.

A number of authors have observed the formation of In–H pairs in silicon [24–26] and germanium [25]. In silicon, two In–H complexes are known to form, with $\langle 111 \rangle$ axial orientation [24], thought to be In bonded to a single hydrogen in either a neutral or a negative charge state [26]. Only one stoichiometry of this In–H complex was shown to be formed [26]. Assuming In on a substitutional site, the hydrogen has been hypothesized to lie on either the bond-centred (BC), anti-bonding (AB) or tetrahedral (T) sites, in order to give the observed $\langle 111 \rangle$ symmetry. These pairs were found to dissociate at approximately 420 K with an energy of 1.3 eV. The population of these complexes was found to depend on the Fermi level and the concentration of free holes [26]. In germanium only one In–H complex was found to form, also with $\langle 111 \rangle$ symmetry [25]. An upper limit for the dissociation of this complex was set at 1.1 eV. In order to discover whether similar interactions occur in diamond we pre-implanted hydrogen into two of the diamond samples under study.

2. Method

A suite of four natural diamonds was used in the measurements: a pure type IIa stone, a type IIb stone and two pieces cleaved from the same type IaAB stone. The samples were characterized by IR spectroscopy. The IaAB samples were found to have nitrogen in two forms: A centres at $\gtrsim$800 ppm and B centres at $\gtrsim$1100 ppm. The IIb sample was found to have a much smaller nitrogen concentration: A centres = 8.3 ppm and B centres = 4.2 ppm. Type IIb diamond is natural p-type semiconducting diamond containing boron. One of the IaAB samples as well as the IIa sample were pre-implanted with 10 keV H ions at a dose of 5×10^{14} atoms cm^{-2}. Thus we have a suite of samples with a large number of various defect configurations, including boron, hydrogen, nitrogen in different complexes and vacancies. It should be remembered that all the samples do contain extended defects in the form of dislocations and grain boundaries, and sub-microscopic hydrogen-rich inclusions from the parental magma, as is common to all natural diamonds. The IIa stone was polished away from any major plane, such that the [110] and the [111] axes were both less than 35° from the surface normal. The other stones were all cleaved and then polished as close to the (111) surface as possible (within a few degrees).

All the samples were implanted with 60 keV ^{111}In ions at the on-line mass separator, ISOLDE. Due to constraints on beam-time and current the samples were implanted at a range of doses, from 1.59 to 6.04×10^{12} atoms cm^{-2}. This is approximately four orders of magnitude below the amorphization threshold for In in diamond [28]. This is the point beyond which irreversible amorphization of the diamond lattice occurs. The implantations were all performed at room temperature, through a 1 mm collimator. All implantations were made 7° off-axis in directions chosen to avoid channelling effects down any major axes or planes. Using the TRIM98 Monte Carlo simulation code [29], the vacancy distribution due to the implanted indium ions was calculated. The In depth distribution was found to be peaked at 188 Å with a maximum concentration of between 9 and 35 ppm (atomic), depending on the dose. The vacancy concentration at this depth was calculated to be on average for the samples approximately 1150 ppm. This high concentration is due to the fact that each In ion is calculated to create 273 vacancies in the diamond bulk. The hydrogen implantation of two of the samples results in a hydrogen profile which is peaked well beyond that of the In, at 689 Å. This is due to 10 keV being the lower limit on the energy obtainable from our ion implanter.

Using TRIM98 the hydrogen concentration at the peak of the In implantation range profile was found to be approximately 14 ppm (atomic), although the concentration varies somewhat throughout the In distribution. The samples implanted with hydrogen have an average increased vacancy concentration of approximately 1400 ppm at the In peak. This vacancy distribution also extends further into the bulk than that due to the In ions alone. TRIM98 simulations of the implantation profiles are shown in figure 1.

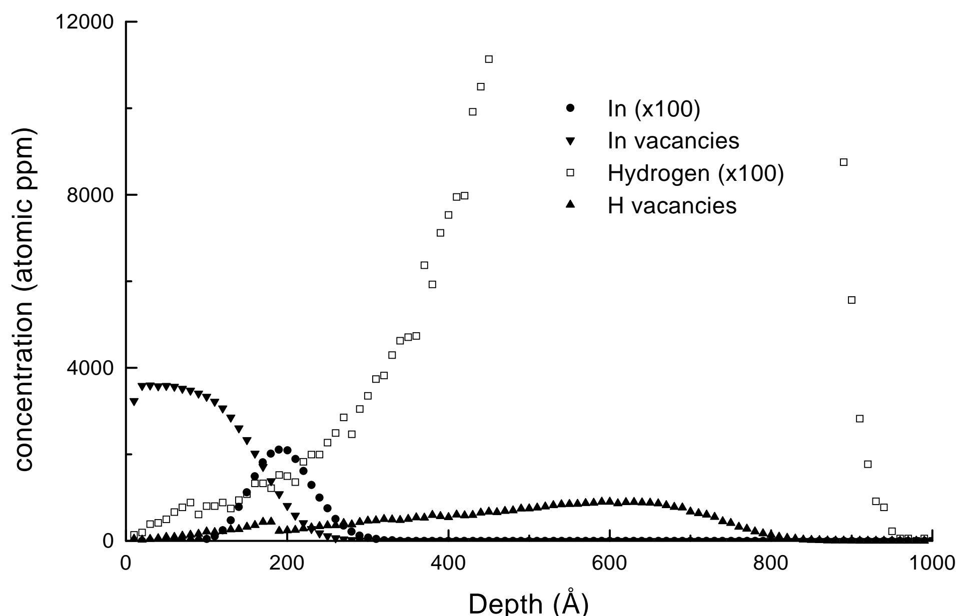


Figure 1. TRIM98 Monte Carlo simulations of the resultant indium, hydrogen and vacancy distributions after implantation. The indium dose in the simulation was 3.8×10^{12} atoms cm^{-2} and that of the hydrogen was 5.0×10^{14} atoms cm^{-2}. From approximately 450 to 900 Å the hydrogen distribution extends upwards beyond the range of this plot.

All the samples were annealed at 1373 K for 20 minutes in a vacuum of 1×10^{-6} Torr, before measuring. This is above the value for the second stage of the In-in-diamond annealing cycle [8, 9, 11]. The exception was the IIb sample which was measured both pre- and post-anneal. Measurements were performed in all major axial directions, apart from for the IIa sample where measurements were not made in the [100] direction due to the sample geometry. Each measurement took roughly between eight and twelve hours, depending on the resident sample activity.

The emitted electrons were detected with a 22×22 Si pad detector developed at CERN, originally for use in particle physics [30]. The detector was situated at a distance of 285 mm from the sample, allowing a channelling pattern of $6^\circ \times 6^\circ$ to be covered. The individual pads have an area of 1.3×1.3 mm^2. This detector has a resolution of approximately 5.2 keV at the electron energies of interest, i.e. between 145 and 242 keV. The angular resolution of the detector was calculated, using the method outlined in [31], to be approximately 0.1°. This is due both to the position resolution of the detector and the finite size of the implanted beam-spot. Further information on the particular detector used in these measurements can be found in [32]. There are numerous benefits in two-dimensional CEEC to using a position-sensitive detector (PSD) over conventional CEEC. These include an increase in detector efficiency by about two orders of magnitude which allows one to better sample the rich structure of the two-dimensional

channelling spectra [32], enabling more detailed information on the occupied lattice sites to be obtained. One-dimensional channelling suffers from the fact that planar channelling effects are often not considered, and that one-dimensional line-scans inevitably cross planes. This yields uncertainty in the normalization of the measured channelling yield.

The data were fitted using the theory of electron diffraction in the many-beam formalism, using non-linear least-squares fitting routines. A review of this can be found in [33]. Simulations of channelling patterns generated for various sites can be seen in figure 2.

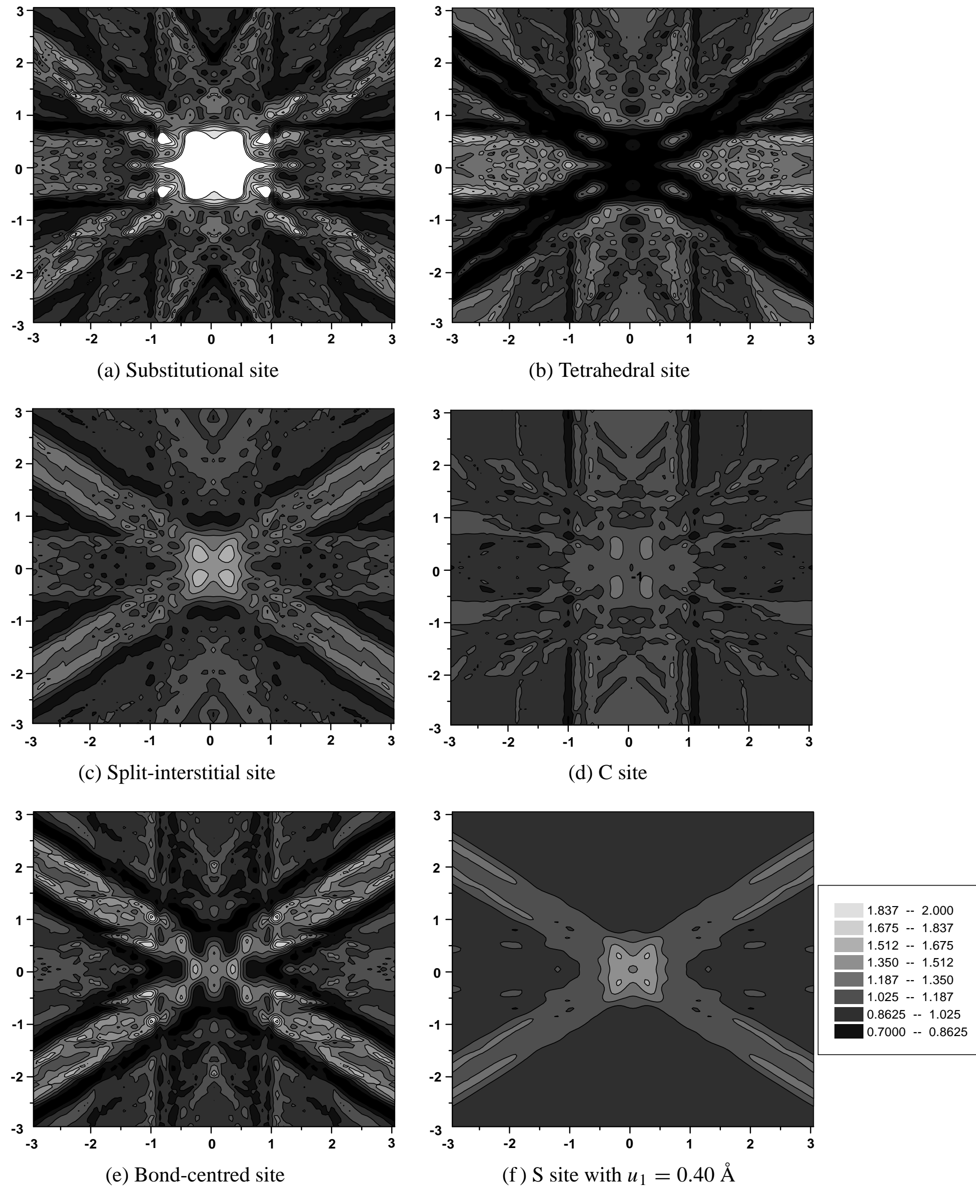
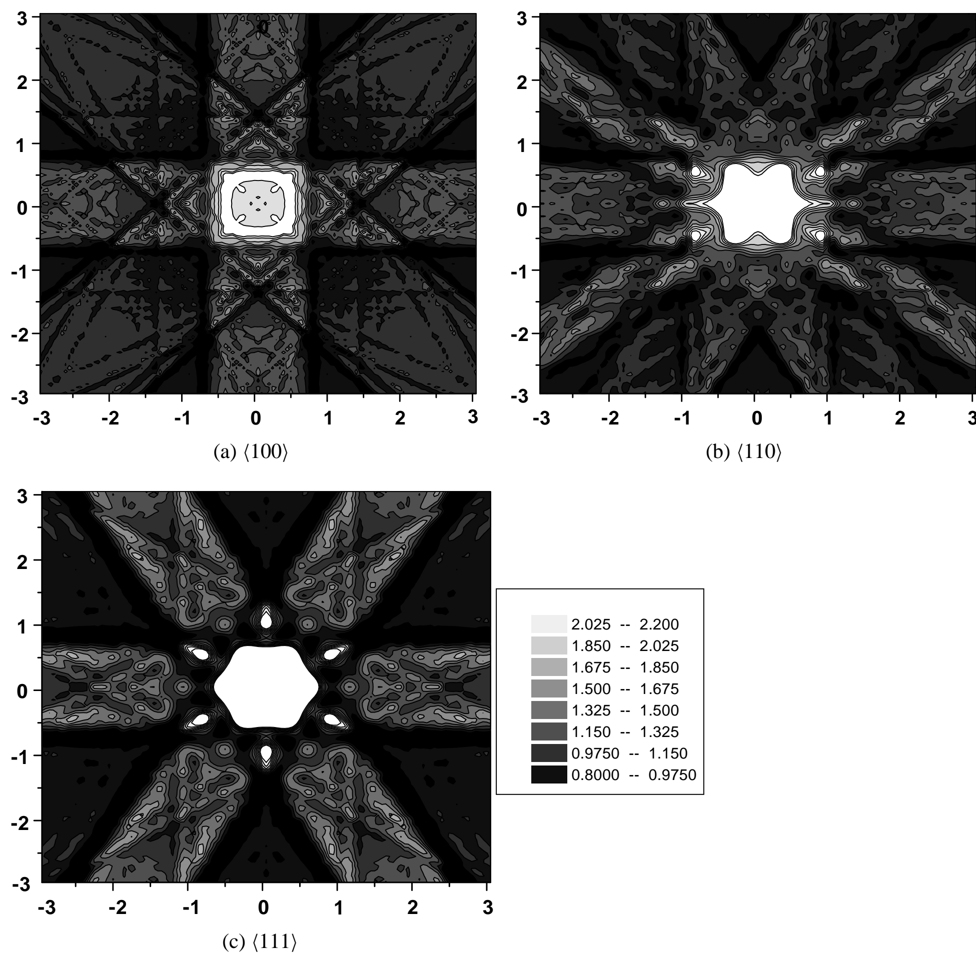


Figure 2. The calculated different channelling patterns obtained for emitters at different lattice sites, as seen down the [110] direction (for a given emitter depth). Panel (f) is a substitutional site with a vibrational amplitude of 0.40 Å.

These simulations are for a pure diamond structure and are thus independent of diamond type. The simulations were folded with the experimental angular resolution, which washes out the fine structure due to quantum mechanical diffraction. This can be seen by comparing the theoretical simulations in figures 2 and 3 with the calculated fits in figures 4 and 5. The sites considered in the simulations were substitutional, tetrahedral, bond-centred, anti-bonding, hexagonal, split-interstitial and the C and Y sites. Various points between these along the ⟨111⟩ and ⟨100⟩ directions were also fitted. The C site is the site midway between the substitutional and tetrahedral sites in the ⟨100⟩ direction, and the Y site is that between the C site and the tetrahedral site on the same axis. The projected position of a site, when viewed down a particular channelling direction, is not always the same for the different equivalent directions in the crystal lattice. This degeneracy in apparent position was accounted for by linear weighting of the different equivalent positions for each site in each channelling direction considered. Vibrational amplitudes of both the In emitter atoms and the carbon atoms of the host lattice were empirically determined from tabulated Debye temperatures in the literature [34, 35]. The Debye temperatures used were 1860 K for diamond and 612 K for the In in diamond.



(a) ⟨100⟩

(b) ⟨110⟩

(c) ⟨111⟩

Figure 3. Calculated channelling yield for a substitutional emitter (at a given depth), as seen along the three major axial directions.

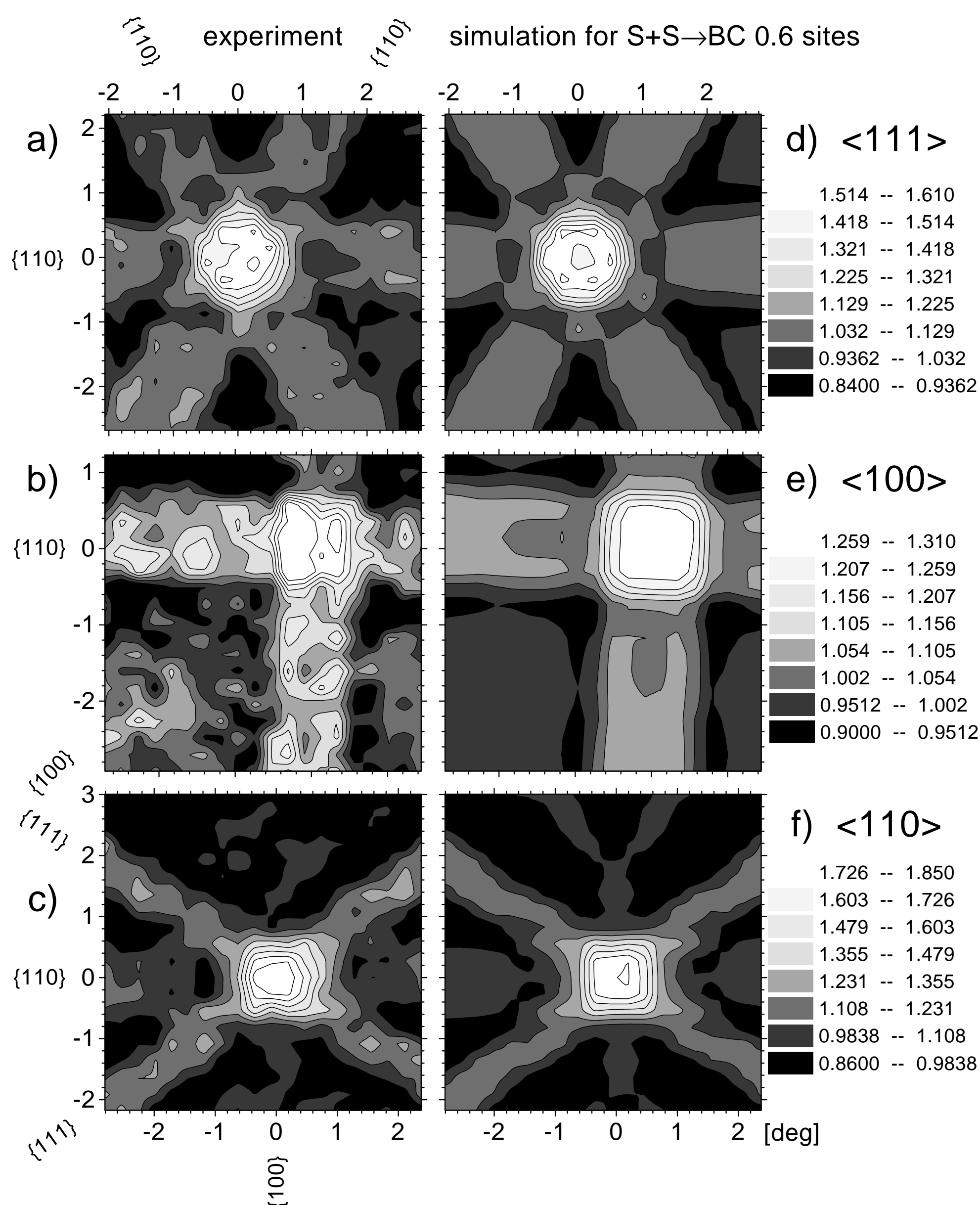


Figure 4. Calculated fits to the experimental data for the IIb post-anneal sample in all three major axial directions, using a combination of substitutional and S–BC(0.6) sites. The experimental data are in the first column and the corresponding calculated fits in the second.

The expected value for the In vibrational amplitude was 0.034 Å. This parameter was varied for the substitutional site up to a value of 0.400 Å in order to try to improve the fit. In our vibrational model this is equivalent to the In occupying an isotropic distribution around the substitutional site. A detailed description of the fitting procedure can be found in [32]. Combinations of the best fits were then made (up to a maximum of three sites being present in the simulation), to find the best overall fit. The calculated channelling patterns for different

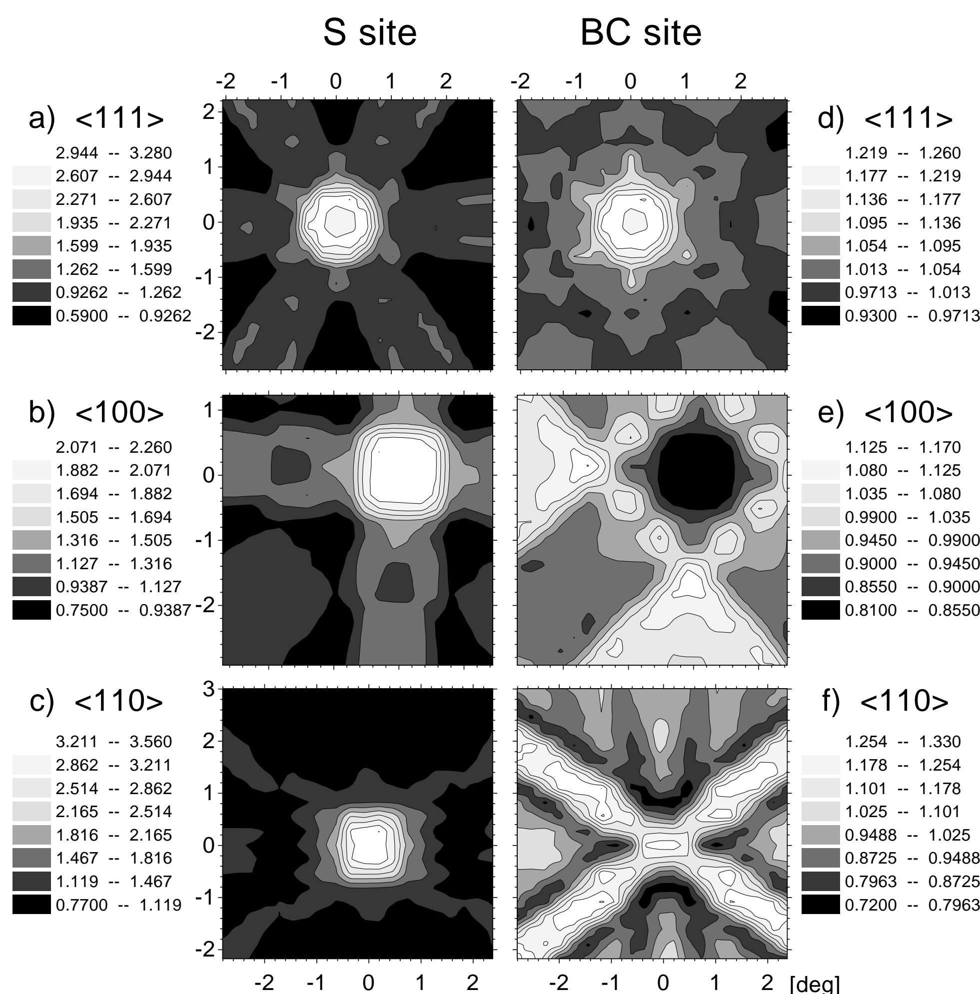


Figure 5. Calculated fits to the experimental data for the IIb post-anneal sample using firstly only a substitutional fraction, and secondly only a bond-centred fraction. Neither yields as good a fit as the substitutional plus S–BC(0.6) combination.

axial directions for an emitter located at a substitutional site are shown in figure 3. The best fits were then compared with the other major channelling directions to find the sites occupied and the relevant fractions. The fractions were corrected for background in the energy spectra by fitting a trapezoidal function to the background components under the relevant energy peaks. It should be noted that in the case of small displacements from a given site the analysis gives the *mean* displacement of the probe atom. Thus it is not possible to distinguish between an ensemble of emitters with the same discrete displacement or an ensemble with a small distribution of values around that same value [32].

3. Results and discussion

The fractions obtained for the best fits to the experimental data are shown in table 1. The most favoured sites were a combination of a substitutional (with a 0.034 Å vibrational amplitude)

Table 1. Site fractions obtained for ^{111}In emitters implanted into various diamond samples.

Sample	Substitutional	S → BC(0.6) along ⟨111⟩	Random
IIb—pre-anneal	12(10)%	55(10)%	33(8)%
IIb—post-anneal	31(5)%	45(3)%	24(3)%
IIa (hydrogen implanted)	22(8)%	54(17)%	24(8)%
Ia	28(6)%	59(8)%	13(8)%
Ia (hydrogen implanted)	29(4)%	58(4)%	13(9)%

and a site approximately 60% of the way (∼0.45 Å) from the substitutional to the bond centre along the ⟨111⟩ axis (which we shall refer to as S–BC(0.6)). Examples of fits against experimental data are given in figure 4 for the IIb post-anneal sample. The errors are determined by the differences in fractions obtained for a sample from the data acquired using the different channelling directions. Such differences are always present due to errors introduced by the dechannelling approximation in the simulations, errors in the In implantation depth profile in a single crystal, as well as by the imperfect treatment of the angular resolution. In previous results the majority of In atoms were found to favour near-substitutional sites. We have been able to distinguish two distinct fractions, one the ideal substitutional sites and the other the S–BC(0.6) sites. This new insight is due to the added information in the two-dimensional data collected. Combinations of pure substitutional and random fractions (as in previous work) were fitted, but did not yield as good a fit, as shown in figure 5 (the plots of the experimental data against the calculated fits for the diamond types have not been shown purely due to space constraints). The reduction in planar (100) channelling effects seen in the data is also a good indication of a population of the emitters moving off-substitutional. Within the errors the fractions obtained are seen to be nearly identical for all samples, independent of defect concentration. This does not exclude specific In–defect complexation with boron, nitrogen or hydrogen. Rather, if such complexation does occur, it does not affect the In lattice site enough to influence its channelling spectra; i.e. less than 0.1 Å displacement of the In lattice site from sample to sample takes place. The tendency for a significant fraction of the In to be off-substitutional concurs with earlier observations of defects near the In lattice site [8, 10], which have the effect of breaking the symmetry of the lattice, thus generating an off-centre equilibrium In position. However, in order for the S–BC(0.6) site to exist, some sort of defect must be present to create this asymmetrical configuration. No theoretical modelling of this site has ever been performed. A distinct possibility is that the In forms complexes with implantation-induced defects involving vacancies, self-interstitials or possibly both. These could then dominate the In–defect formation process. It is known that large ions which are not charged (and thus the Coulombic interaction is absent) are prone to attract vacancies through elastic interaction, whereas small ions attract self-interstitials [36]. Therefore In–vacancy complexes are probably favoured over self-interstitial ones. In the case of the IIb sample the difference in Fermi level is seen not to have a measurable effect. In silicon, Cd–H pairs (the method used measured the structural properties after the transmutation from ^{111}In) were found to exist in different charge states, the populations of which were found to depend on the Fermi level and the free-hole concentration [27]. The charge state was implied not to affect the In/Cd lattice site.

We now consider the vacancy–In–vacancy complex for which calculations were made in previous work [12]. The off-centre substitutional site in that work is a bond-centred site between two vacancies. To first order we treat this site purely as bond centred. This is because the emitted electrons in the CEEC technique are relatively insensitive to the local atomic configuration and are dominantly dependent on the geometric location of the emitter atom.

The bond-centred site was tried in the fitting procedure and was found not to fit the data, except as a very small fraction (∼2%), in combination with other sites. Fits to the experimental data for one of the samples using only a bond-centred fraction can be seen in figure 5. A likely configuration is the In in a divacancy (as in the calculated system), with a vacancy or interstitial at a next-nearest-neighbour position or the like, which breaks the symmetry, allowing the In to relax along the [111] direction. More than two vacancies being present in the system was not considered in the theoretical calculations and is a possibility, due to the large number of vacancies introduced in implantation.

The effect of annealing the sample can clearly be seen in the IIb pre- and post-anneal fractions. Upon annealing, the fraction of emitters on substitutional sites increased as expected, at the expense of the emitters at both the S–BC(0.6) and the random sites. This is in agreement with the previous work done on this system [8, 9, 11]. Since annealing is known to heal the lattice through the recombination of vacancies and self-interstitials, this suggests that In at the S–BC(0.6) site involves these defects, and that In at the substitutional site does not. After annealing, recombination of vacancies and self-interstitials decreases this fraction.

As mentioned earlier, previous measurements have yielded a random or irregular fraction, the origin of which is unknown. The random fraction could (partially) contain In bonded in various different multi-vacancy complexes, which yield a variety of different low-fraction In lattice positions. The random sites are those of low enough concentration (less than a few per cent of probe atoms occupy them) that they cannot be individually distinguished. If complexation with boron, hydrogen or nitrogen occurs at a very low level (less than a few per cent), then this would also form part of the random fraction.

The existence of a substantial substitutional fraction, in both these and previous measurements, is perhaps harder to explain. In the theoretical work [12], substitutional In was found to be at best a metastable state. Ballistic considerations for just slightly above thermal equilibrium near the end of the implantation cascade may lead to this configuration. Annealing may restore damage in such a way as to increase the substitutional fraction.

The lack of any noticeable effect due to the formation of In–H pairs can be correlated to similar work on silicon [27]. In this study the number of such pairs formed was found to have a strong dependence on the implantation energy. The number of In–H pairs for a 200 eV H^+ implant was found to be approximately ten times greater than if the implant was done at 1 keV. This was postulated to be because of vacancies caused during implantation (at 200 eV fewer than 0.5 vacancies per ion are created, whereas approximately 1.15 per hydrogen ion are created at 1 keV). The vacancies were thought to provide strongly competitive traps for the hydrogen. It should be noted that the detection of In–H pairs in silicon was via the technique of perturbed angular correlations (PAC), not CEEC. PAC measurements on In in diamond found no complexation with hydrogen [37]. Studies on hydrogen implantation into diamond at similar energies and doses to those used in this work have shown it to strongly trap in its own range distribution [38–40]. We expect this to have been the case in our study.

4. Conclusions

Previous studies by other groups on In in diamond have been extended to include a number of defect-rich systems. This comprehensive two-dimensional work is a significant improvement over earlier measurements. The results indicate that the In finds itself either at a defect-free substitutional position, at a position between substitutional and bond-centred sites (60% towards the BC), or at various other low-fraction, irregular sites which may involve vacancies. The S–BC(0.6) site is thought to be a divacancy plus another, more distant defect. The previously determined substitutional or near-substitutional fraction has thus been

differentiated into an exactly substitutional site plus one S–BC(0.6) approximately 0.45 Å away. Complexation with any of the defects, including boron (p-type), hydrogen and nitrogen, although not excluded, is not detected.

The importance of vacancies, single and multiple, produced during implantation, for the final In–host configuration in diamond has been suggested in this paper. Future work should concentrate on this defect which in many cases is thought to dominate over other impurities, whether intrinsic to the sample or introduced into it during implantation. It is thought to play a major role in the random fraction detected in these and earlier measurements. Theoretical modelling of impurities such as In in diamond should be extended to include single- and multi-vacancy configurations. Further experimental work is needed to confirm the nature of the sites in question.

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