High resolution Laplace deep level transient spectroscopy of p-type polycrystalline diamond

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1 Introduction There has been considerable interest for many years in the use of diamond as a semiconductor particularly for high power electronics and detectors. Diamond is predicted to have outstanding mechanical, chemical and electronic properties, due to its very strong sp^3 bonding [1] and excellent resilience to harsh environments. It has a very wide band gap, about 5.5 eV, and can be doped n-type or p-type. However, the dopant ionisation energies, especially in polycrystalline diamond, are rather large, about 370 meV for B, a p-type dopant, and between 0.56 eV and 1.7 eV for n-type dopants. The result of this is that conductivity is low at room temperature. Doped semiconducting diamond can be grown by a variety of Chemical Vapour Deposition (CVD) methods, and each method results in different types of diamond films. Some of the properties that may differ include the diamond particle size, grain boundary nature, hydrogen content, and the defect density [2] which includes point and extended defects. Vacancies, interstitials, and combinations of dopants and de-

fects give rise to point defects, which may have associated electronic levels in the band gap, and grain boundaries and dislocations are the causes of the extended defect population. Defect densities are thought to be high in polycrystalline diamond, due to the non-homogeneous structure of the films. This has consequences for device operation, since for example, in the case of diamond detectors there is much in the literature that suggests that a deep trap level has to be filled in order to improve the device sensitivity [3−5]. Monocrystalline diamond overcomes some of these problems but availability is still limited, and the material may have some reproducibility problems [6]. Large-scale production of single crystal devices based on homoepitaxial growth is still thought to be unfeasible because the sample sizes are relatively small, due to the requirement for a single crystal seed, and growth rates are fairly low. Moreover, it has recently been reported that large grain polycrystalline diamond can exhibit better transistor properties, in terms of higher drain currents and threshold fre-

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quency, than single crystal material [7]. Therefore there is still a need to understand and optimise polycrystalline diamond performance.

In a material where free carrier concentrations are low at room temperature because of large dopant ionization energies, the presence of electrically active defects is highly undesirable as they can trap and therefore further reduce the number of free carriers. The de-trapping process also causes leakage currents. Thermally stimulated current and thermoluminescence measurements in detector material have shown that these processes are occurring, and very deep levels have been observed, between 1.1 eV to 1.8 eV [8, 9]. Such measurements are not able to distinguish between hole or electron emission though, and they do not reveal which carrier band is involved in the trapping or emission process. Capacitance Deep Level Transient Spectroscopy (DLTS) is able to do this, but is notoriously difficult on wide band gap materials, as the geometric capacitance of the sample is quickly reached, when the applied voltage does not influence the depletion region width. However, if a capacitance–voltage measurement can be carried out, where the value of C is proportional to the applied voltage, then the device is a candidate for capacitance DLTS.

Recent work, using a combination of techniques, has shown that trapped charge at grain boundaries can dramatically alter the electric field profile within poly-CVD particle detectors and therefore their performance [10]. The use of capacitance DLTS can enhance these types of studies in many ways. Firstly, the carrier involved in the trapping process can be unambiguously identified, secondly, the capture cross-section of the defect can be directly established, and finally, DLTS can be used to identify the differences between point and extended defects. Though this is routinely done in Si [11], it has also recently been applied to other, more complex, wide band gap semiconductors [12]. The test for the spatial nature of the defect is based upon directly measuring the defect capture cross sections: if the capture rate at the defect is time dependent, the defect is not point-like, as charge capture at the defect results in the build up of a Coulombic, repulsive barrier, which slows further capture [13].

A high resolution modification of DLTS, Laplace DLTS [14], is capable of directly establishing whether traps have single carrier emission rates or not i.e. whether the DLTS peak is due to emission from a point defect, or many defects closely spaced in energy. In this technique, the capacitance transient is measured at a fixed temperature. It is then inverse transformed, and one or more emission rates show clearly in the output. More than one emission rates in the transient is often indicative that an extended defect is capturing and emitting carriers. To the best of our knowledge, this technique has not yet been applied to semiconducting diamond. In this paper, DLTS and high resolution technique Laplace DLTS are applied to semiconducting, boron doped CVD diamond grown on Si, and a combination of the measurement techniques is used to demonstrate whether the electrically active defects present in the material are point or extended defects.

2 Experimental details The boron doped polycrystalline diamond films were grown by hot filament chemical vapour deposition at a process pressure of 20 Torr using 1% CH_4/H_2 as the process gases plus B_2H_6 as a variable but controlled gaseous source of boron. The single crystal (100)Si substrates were manually abraded before deposition using $1-3$ μm diamond grit. Rhenium was used for the filament material since Re does not act as a sink for carbon or boron species, unlike Ta which can absorb significant amounts of B and C for many hours into the deposition run [15]. The growth time was 8 h, giving a diamond film thickness of ~4 μm. Film morphology was polycrystalline with facetted crystallite sizes averaging ~1 μm in size.

The boron concentrations in the measured diamond ranged from 7×10^{18} cm^{-3} to 2.1×10^{20} cm^{-3}. The substrates were n and p-type Si, with doping levels of 3×10^{15} cm^{-3} and 8×10^{16} cm^{-3}, respectively. The diamond surface was oxygen-terminated before any processing. The diamond on n-type Si formed a p–n diode and Ohmic contacts were fabricated on both the Si and the diamond. Al was evaporated on the Si back contact while the diamond Ohmic contacts were formed by sputtering Ti:Pt:Au through a patterned mask of 1 mm dots. The diamond on the p-type Si had Al Schottky contacts evaporated through the patterned mask and Al Ohmic contacts were evaporated at the back of Si.

Capacitance DLTS on the p–n diode was carried out at a range of fill pulses. The first fill pulse was negative, to ensure that little minority carrier injection occurred across the junction, and the fill pulse bias was then increased in steps of 1 V to uncover any effects of minority injection, until the diode was fully turned on. The DLTS on the Schottky diode was carried out with a small forward bias, as is the normal practice for this measurement.

In other reported work it is quite usual to use a form of DLTS that measures charge in an external circuit, which is entirely appropriate for insulating and less conducting samples, such as discussed in [16]. However our samples were sufficiently conducting to allow capacitance DLTS.

The LDLTS was then carried out at fixed temperatures in a high stability cryostat. The temperature was chosen by examination of the DLTS spectrum: LDLTS measurements were taken at temperatures where there was a DLTS peak, i.e. where the emission rate of the carriers matched the rate window. In LDTS several thousand capacitance transients are averaged. This ensures that the signal-to-noise ratio is of the order of 1000, which is necessary to separate transients with closely spaced emission rates. The transient was then analyzed by one of three available inverse Laplace transform mathematical algorithms, and a plot of peak intensity as a function of emission rate produced. If the transient is truly exponential, only one peak is visible in the LDLTS spectrum. The area under each sharp peak is

proportional to the trap concentration. Multiple peaks are indicative of either closely spaced electronic levels, or the presence of extended defects. The technique has been applied extensively to separate closely spaced point defects in Si for many years [17], but has since been extended to wider band gap materials [18].

3 Results

3.1 Carrier concentrations Details of the B and carrier concentration of the Schottky and p−n diodes investigated with LDLTS are presented in Table 1. The doping concentration was measured by SIMS while the carrier concentration was measured from capacitance−voltage measurements. Due to the size of the diamond bandgap and the large activation energies of its dopants, only a fraction of the doping population is ionised at room temperature and this may explain the difference between the doping and carrier concentrations in the table. In addition, it has been established that the B acceptor level approaches the valence band for high doping concentrations leading to hopping conduction [19, 20]. In particular, for B doping higher than 3×10^{20} cm^{-3} it is believed that a phenomenon similar to metallic conduction leads to the majority of the B being ionised, due to the overlapping of the acceptor level and the valence band.

Diamond films with higher B contents than those in Table 1 were also investigated. These contained B doping of 7.6×10^{20} cm^{-3} for a Schottky and 7×10^{21} cm^{-3} for a p−n diode, which is well in the range of metallic conduction, and, as expected they did not yield any DLTS spectra. Nonetheless, their investigation was interesting for two reasons. Firstly, it was confirmed that metallic conduction is indeed present in polycrystalline diamond. Secondly, in the case of the p−n diodes, where the very high diamond doping ensures that the depletion region lies entirely in the Si, the fact that no DLTS signal was observed from the diamond on n-type Si verifies that the observed traps in the lower doped diodes S4 and S5 are due to defects located in the diamond and not in the Si. The diamond in diode S4 was kept intentionally undoped though it did exhibit conductivity, therefore in this sample the depletion region lies both in the diamond and in the Si. Thus it was important to establish that there were no defects attributable to the Si.

3.2 Schottky diodes The DLTS results for diode S1 with a −2 V reverse bias, a 1 V fill pulse and a 1 ms fill pulse length are shown in Fig. 1 for rate window 50 s^{-1}.

Table 1 Doping and carrier concentrations of diodes investigated.

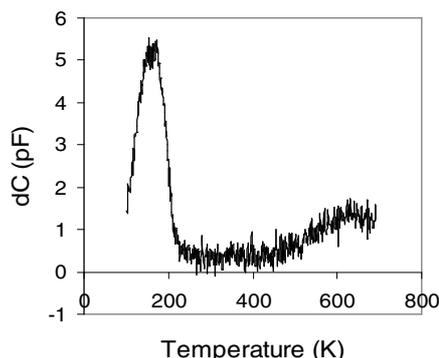|		B doping concentration (cm^{-3})	carrier concentration (cm^{-3})
Schottky diodes	S1	1.9×10^{20}	1.5×10^{18}
	S2	2.1×10^{20}	6×10^{18}
p−n diodes	S4	background	5×10^{15}
	S5	7×10^{18}	6×10^{15}

Figure 1 DLTS spectra of Schottky diode S1 with rate window 50 s^{-1}, reverse bias −2 V and fill pulse 1 V.

Diode S2 exhibited a similar spectrum. There is a well defined defect level at about 170 K for both diodes and a very broad spectrum starting around 400 K. The broad, higher temperature peak has an activation energy of approximately 1.06 eV.

The defect at 170 K was found to have activation energies of 0.23 eV for sample S1 and 0.21 eV for sample S2 using conventional DLTS results to obtain the Arrhenius plot. The DLTS feature indicates that this could be a point defect due to its symmetry and location on the temperature scale and there is no clear indication that it may contain multiple emission rates. Capture cross-section measurements (not shown) however, revealed an exponential capture with time, suggesting this may be an extended defect. The high resolution LDLTS should therefore be able to distinguish one or more closely spaced energy levels which are commonly associated with the presence of an extended defect. LDLTS measurements were performed under the same biasing conditions as DLTS, not only at the temperature of the maximum of the peak but over the entire temperature range that the DLTS feature spanned. The measured emission rates are shown in Figs. 2 and 3 for S1 and S2 respectively, at 160 K and 200 K for comparison. Both Figs. 2 and 3 show that there are three distinct emission
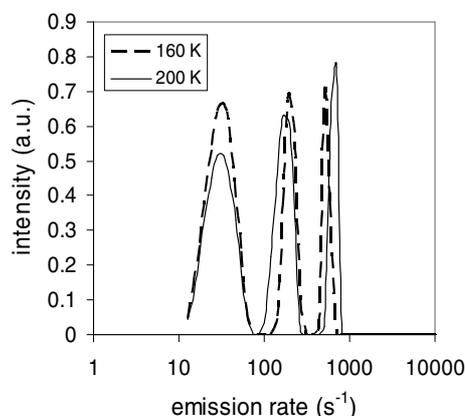

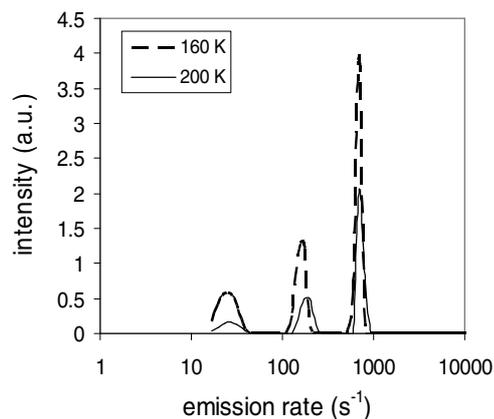**Figure 2** LDLTS of S1 at 160 K and 200 K with reverse bias −2 V, fill pulse 1 V and fill pulse length 1 ms.

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Figure 3 LDLTS of S2 at 160 K and 200 K with reverse bias −2 V, fill pulse 1 V and fill pulse length 1 ms.

Figure 4 DLTS spectra of S4 and S5 with 200 s^{-1} rate window.

rates, confirming that this defect possesses three closely spaced electronic levels in the gap. The appearance of the same number of emission rates in both samples means these are unlikely to be artefacts in the measurements. Furthermore we obtained the same results with three different algorithms used, CONTIN, FLOG and FTIKREG.

For both diodes, the three emission rates at around 30, 180 s^{-1} and 680 s^{-1} are covering a fairly broad spectrum of emissions. From Figs. 2 and 3 it can be seen that changing the temperature does not particularly influence the emission of these traps. For distinct levels the emission rate is known to increase when the temperature rises as more energy is provided to the system due to heating. To investigate this effect, LDLTS was performed on these diodes from 80−240 K in steps of 10 K. Firstly, it was observed that the three rates of Figs. 2 and 3 are always present in all these measurements as already indicated by the results at 160 K and 200 K. Secondly, the emission rate varied only a little, or not at all, with temperature. Figure 3 shows no variation for both the low emission and high emission rate and it is clear that the low emission rate of Fig. 2 does not vary either.

Due to the high resolution of the LDLTS measurement an accurate calculation of the activation energy of a trap can often be performed. This is achieved by recording the emission rate of the trap as deduced from the inverse transform of the measured capacitance, as a function of the temperature, for a temperature range approximately covering the feature width in the original DLTS measurement. By plotting the results in a normal Arrhenius diagram, the activation energy can be calculated with considerable accuracy.

This was attempted for the three emission rates of S1 and S2. Because the emission rates did not vary consistently with temperature, calculation of the activation energy in both cases did not yield meaningful results. However, this conclusion is an indication of the presence of extended defects, since the emission or capture of carriers at extended defects is time dependent. Consequently it is concluded that the level observed by DLTS at 170 K is due
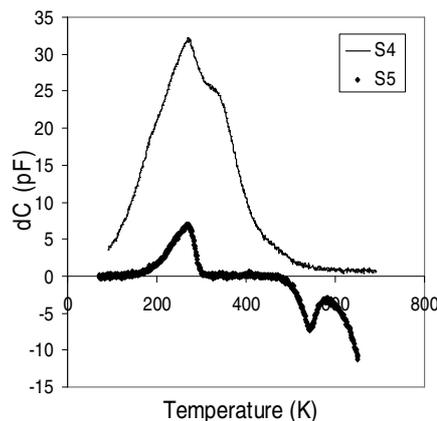
to an extended defect, at which carrier capture causes a local change in the band structure meaning that the activation energy cannot be deduced. A direct measurement of the capture cross section (not shown) supports this suggestion.

3.3 p−n diodes The DLTS of the p−n diode structures, samples S4 and S5, revealed a broad defect spectrum covering a temperature range from 100−400 K. Figure 4 shows the DLTS when a fill pulse of 2 V was applied, which saturates the hole traps in the diamond because the diode is fully turned on. Two majority peaks were observed for sample S4, and a calculation of their activation energies yielded 0.16 eV for the low temperature level and 0.35 eV for the higher temperature one. S5 exhibited one majority and one minority carrier peak.

The activation energy of the majority carrier trap as found from DLTS was 0.11 eV while the minority carrier trap did not yield a meaningful activation energy as the peak temperature increased for smaller rate windows. LDLTS was applied to both samples at a temperature range that encompassed the majority peak.

Figure 5 shows the LDLTS of S4 measured with the same biasing conditions as the DLTS in Fig. 4. Data was collected at a wide range of temperatures, and data at 180 K and 350 K are shown in Fig. 5 as two examples. The emission rates change with temperature and the intensity of the emission spectra at 180 K is much larger than that recorded at 350 K. In particular the high emission rate at 180 K has been scaled down by 50 times in order to facilitate a comparison between the two temperatures. From 130 K until 300 K the high emission rate is larger than the other two rates and has a highest emission rate of around 4000 s^{-1}. After 280 K however, its emission rate starts to reduce as well as its intensity, as shown in Fig. 6. Above 300 K its emission steadily reduces until the temperature reaches about 410 K. Because the emission rates of these defects do not reduce with reducing temperature, meaningful activation energy calculations could not be performed. However, the fact that three emission rates are detected throughout the entire examined temperature range, imply
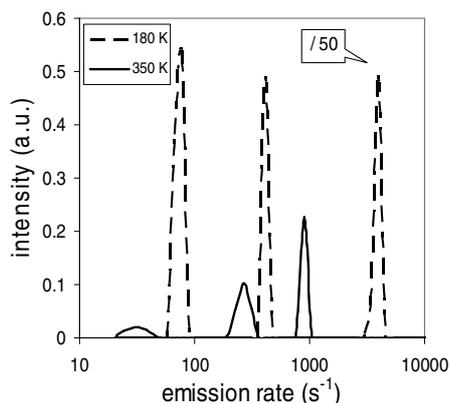
Figure 5 LDLTS results of S4 at 180 K and 350 K with the high emission rate at 180 K reduced by 50 times, as shown in the figure, for ease of comparison.

Figure 7 LDLTS results of S5 at 230 K and 260 K under the same biasing conditions as DLTS.

that at least three defect levels are present. Comparing with the DLTS in Fig. 4 where only two levels were observed in this temperature range, LDLTS has demonstrated the improvement in resolution that this technique can offer.

The LDLTS results of S5 are illustrated in Fig. 7. From 300 K and as the temperature reduces to 250 K one emission rate is detected (peak A in Fig. 7). The emission rate of this peak reduces with temperature throughout the entire range 300–200 K. However, at 240 K another peak appears whose emission rate also consistently reduces with temperature over the range 240–200 K (peak B in Fig. 7). Therefore activation energy calculations were possible for this diode.

The activation energy of the emission rate in peak A between 300–200 K was found to be 0.21 eV while the activation energy of the defect appearing at 240 K (peak B) and was 0.1 eV. Because capacitance DLTS is used, it can be unambiguously stated that these are hole traps, and also that the negative going excursion in Fig. 4 for sample S5 means that it also contains electron traps. Charge DLTS is unable to make this distinction.
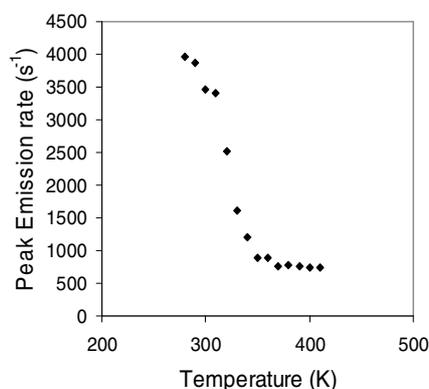
4 Discussion Two types of structures have been characterised in this work: p-type diamond on p-type Si on which a Schottky diode is formed, and p-type diamond on n-type Si, which forms a p–n diode. The DLTS and LDLTS therefore samples different regions of the devices. The experiments on the Schottky diodes sample a volume near the surface whilst the p–n diode experiments sample a region nearer to the diamond-Si interface. It is well known that the extended defect profile of polycrystalline diamond changes as growth proceeds, and therefore we may also expect to see an associated changing profile of electrically active defects. Capacitance DLTS on the Schottky diodes, revealed a level around 0.2 eV, which is in good agreement with charge-DLTS (Q-DLTS) measurements of undoped or lightly B-doped polycrystalline diamond films grown by HFCVD previously reported [21, 22]. The fact that three emission rates were consistently detected in our LDLTS measurements of the Schottky diodes, may perhaps be an indication that this activation energy is not due to a discrete level as was so far believed [21]. The LDLTS measurements therefore, constitute further proof that non-substitutional B atoms trapped at grain boundaries may give rise to levels with different activation energies, than the substitutional B acceptor level at 0.37 eV [16]. A similar activation energy of 0.21 eV was detected by LDLTS near the interface of diamond with Si in the p–n diodes. However, in this case a more clear emission rate dependence with temperature was observed indicating a well-defined level potentially indicating more point-like defect behaviour. Furthermore, a similar level to the one detected here at 0.1 eV has been previously associated with the presence of hydrogen in MPCVD grown diamond films [23]. Although these samples were oxygen-terminated on the diamond surface, it is very likely that hydrogen is present in larger volumes near the interface giving rise to this level. A second scenario is that these energies reflect the fact that the B may pile up at the grain boundaries and be in differently strained regions of the crystal, forming new deep defect states which can not be approximated by hydrogen-like wavefunctions, hence the range of new activa-

Figure 6 Peak emission rate versus temperature of the high emission rate of diode S4.

tion energies. Finally, DLTS depth profiling could elucidate whether interface states are also present in this signal, and further investigations are underway.

5 Conclusion High resolution Laplace deep level transient spectroscopy (LDLTS) has been used to characterise deep electronic states in the band gap of polycrystalline p-type diamond. The technique shows that apparently narrow and simple DLTS spectra contain multiple emission rates. These are discussed in terms of the dopant being trapped at different sites at grain boundaries.

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