Growth and electrical characterisation of δ-doped boron layers on (111) diamond surfaces

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A plasma enhanced chemical vapor deposition protocol for the growth of δ-doping of boron in diamond is presented, using the (111) diamond plane as a substrate for diamond growth. AC Hall effect measurements have been performed on oxygen terminated δ-layers and desirable sheet carrier densities (~10^{13} cm^{-2}) for field-effect transistor application are reported with mobilities in excess of what would expected for equivalent but thicker heavily boron-doped diamond films. Temperature-dependent impedance spectroscopy and secondary ion mass spectroscopy measurements show that the grown layers have metallic-like electrical properties with high cut-off frequencies and low thermal impedance activation energies with estimated boron concentrations of approximately 10^{20} cm^{-2}, © 2012 American Institute of Physics. [doi:10.1063/1.3682760]

I. INTRODUCTION

In comparison to other group IV semiconductors diamond possesses outstanding intrinsic electrical properties. The potential for high carrier mobilities, coupled with its high breakdown voltage and high thermal conductivity, make diamond a very attractive material for high power and high frequency electronic devices.1 However, diamond’s shortcoming as an electronic material lies in its short lattice constant resulting in few elements being suitable for substitutional doping, and those which are (boron, nitrogen, and phosphorous) display large thermal activation energies.2,3 Boron, the most commonly used diamond dopant, has the smallest activation energy of 0.37 eV at low doping concentrations (<10^{17} cm^{-3}). This value still means that only a fraction of the boron present is activated at room temperature leading to relatively low concentrations of free carriers. Increasing the boron concentration reduces this activation energy, and at ca. 3×10^{20} cm^{-3} the metal-to-insulator transition point occurs and a fully-activated impurity band is formed via the quantum tunneling of holes between neighboring boron acceptor states.4 Unfortunately, as the activation energy of holes decreases so does carrier mobility, not only because of the increased impurity scattering but also due to the onset of a low-mobility, hopping-like conduction. The resulting material is one that has sub-unity carrier mobility and typical sheet carrier densities in excess of that which are readily controlled by a typical field effect transistor (FET).5

A potential solution to this problem of doping diamond is to use a δ-doped boron layer.6 δ-doping consists of heavily doping a metallic conductive channel in diamond on the nanoscale, with the aim of reducing its sheet carrier density while simultaneously enhancing its mobility. In theory, thin δ-doped layers result in the formation of a V-shaped potential well and a 2D hole gas around the boron-doped region, which allows the partial delocalisation of holes into the surrounding high mobility intrinsic diamond;7 leading to the fully activated holes having a higher mobility. In order to achieve a high degree of hole delocalisation, the δ-doped region must fulfill certain specifications. Firstly, the δ-doped region must be substantially thinner than the de-Broglie wavelength of holes in diamond [ca. 10 nm (Refs. 8, 9)] and have an abrupt δ-function-like depth profile with boron concentration transitions shorter than the Debye length in diamond in order to allow the formation of a 2D hole gas.10 Secondly, the boron concentration, and thereof the hole concentration, must be sufficiently dense (>10^{20} cm^{-2}) so that its charge distribution forms a deep V-shaped potential well within which its carriers can populate more delocalised states with higher mobilities,7,11 but not be so dense that any FET subsequently fabricated would require gate voltages in excess of diamond’s breakdown voltage for its control (>4×10^{13} cm^{-2}).5 This results in a requirement of ca. 1 nm δ-layers. Thus, precise deposition of near-atomic layer thicknesses of boron doped diamond are required, but unlike III-V and Si technologies, where molecular beam epitaxy can be used, diamond layers must be grown using the method of plasma-enhanced chemical vapor deposition (PECVD) which does not offer such immediate levels of control over the thickness of deposited layers. Therefore, in comparison to standard diamond PECVD, δ-doping is particularly challenging. Not only must PECVD parameters be optimized for high quality, heavily boron-doped diamond deposition, but the short timescales

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required for ca. 1 nm of diamond growth result in the need for carefully controlled growth protocols being developed.

To date, all attempts at growing single crystal δ-doped boron layers on diamond have used the (100) lattice plane due to the wide availability of high quality diamond films and the reduced lattice strain expected in comparison to the higher density (111) and (110) planes.6,12–15 While ultra-thin doping profiles have been achieved, relatively poor mobility values have been reported to date, the best being considered to be in the region of 20-30 cm²/Vs,10 although no detailed study of mobility values has yet to appear. In the current study the (111) plane is proposed for δ-layer deposition. The authors have previously shown that the (111) diamond plane can support higher doping densities for a given mobility value than diamond (100),16 and the (111) diamond plane has also been found to be advantageous for the realization of superconductivity in diamond;17 similar advantages may be useful in diamond δ-doping. Firstly, the (111) crystal plane has an eight times higher boron incorporation density than the (100) diamond lattice plane,18 which could allow for thinner δ-layers with equivalent boron content. Furthermore, Kitagoh et al.19 have shown how the (111) diamond plane outperforms the (100) plane by at least one order of magnitude at sustaining high boron dopant densities at low doping thicknesses. Secondly, the (111) plane brings the advantage that its carrier concentrations can exceed its boron concentrations, allowing more carriers into the channel without increasing impurity scattering.17 This doping phenomenon may be a consequence of the formation of additional conductive channels; the distortion of the Fermi surface20 or, less desirably, due to a larger effective mass of holes.21 Thirdly, although (111) diamond is usually avoided as an electronic material due to its defective and dislocated crystal structure, thin layers of (111) diamond (<200 nm) can be of relatively high quality in comparison to (100) diamond.19 (111) diamond may come with the disadvantages of greater surface roughness and increased lattice strain from impurities causing increased carrier scattering.22 However, anisotropic strain in (111) diamond grown by CVD may in fact be lower than that of homogeneous strain in (100) diamond.17 This paper addresses the use of (111) diamond surface for the fabrication of boron δ-doped structures using PECVD. Electrical characteristics have been investigated using AC Hall effect and dielectric impedance spectroscopy and secondary ion mass spectroscopy (SIMS) has been used to investigate the boron concentration profiles in doped samples.

II. δ-DOPING (111) DIAMOND

A. Experimental considerations

Heavily doped boron layers are required (ca. 10²⁰ cm−³) with abrupt 1 nm δ-doped profiles. Considering these requirements a microwave PECVD recipe was designed (a) to give a high boron incorporation density via the optimization of gas ratios in the growth plasma and (b) sharp boron concentration gradients with respect to layer thickness via slower diamond growth rates and a revised protocol of gas injections. The PECVD parameters of pressure and power were fixed at 600 W and 80 Torr, respectively, in order to simplify the interpretation of gas mixture changes during the iterative recipe refinement process.

Firstly, boron concentration was considered. The boron incorporation density primarily depends linearly on the borono-to-carbon ratio in the gas phase, (B/C)gas, and from multiple calibration measurements a value of 600 ppm of (B/C)gas ratio was selected to achieve sufficient doping of 10²⁰ cm⁻³, without producing excessive sheet carrier densities. Secondly, the percentage of methane in the gas mixture was considered. To date δ-growth attempts on the diamond (100) plane have necessitated relatively high methane percentages (>3%) because the (100) plane of diamond is less efficient at incorporating boron at low methane percentages.23 The (111) plane does not share this reduced ability to incorporate boron at low methane percentages and as a result lower methane percentages can be used to achieve slower, more controllable, higher quality and less graphitic diamond depositions, while maintaining high boron incorporation densities. A methane percentage of 0.3% methane (with respect to total gas mixture) was selected for δ-growth, which was purposefully set above ~0.15% CH₄ as to avoid hydrogen etching being dominant during δ-growth,24 resulting in a growth rate of ca. 0.015 mm/s and a 60 s growth period for a 1 nm deposition.

Slower growth rates not only result in better quality diamond25 but also allow for more abrupt doping profiles because less diamond is deposited during the transient periods of the PECVD gas mixture when (B/C)gas and CH₄% are not stable. The order of gas injection was then considered to further optimize these transient growth conditions that occur at the edges of the δ-doped region. To date, δ-growth attempts have used a PECVD background plasma of hydrogen and methane with a short injection of a boron containing substance [boron rod, diborane gas, or trimethylborane (TMB)]. In this work, a different protocol has been used that aims to reduce residual boron doping either side of the δ-doped region and reduce boron contamination in the underlying intrinsic diamond epilayer. Instead, methane has been pulsed into a stable plasma of hydrogen and TMB. This method should be advantageous for depositing δ-layers, firstly, because the initial H₂/TMB plasma contains negligible carbon and so no diamond growth occurs, which, in a boron-PECVD chamber, would be contaminated with boron reducing the efficacy of the V-shaped potential well. Secondly, by injecting CH₄ instead of TMB into the plasma, during transient gas mixture periods the (B/O)gas remains at high values (>600 ppm) instead of being proportional to CH₄%. This is particularly important for the second transient step where, in the case of injecting boron into the gas mixture, long-lived boron gas residues in the chamber would produce a lower boron concentration doped ‘tail’ after the δ-layer. Injecting CH₄ avoids this by the CH₄ gas residue only resulting in a slowing growth rate (and eventual slight etching of diamond) in an increasingly high (B/C)gas plasma, which should result in an abrupt boron concentration gradient.

B. Experimental methods

To fabricate heavily boron-doped δ-doped diamond layers, polished 2 mm square (111) diamond substrates (2×2 mm, Suitomo Electric Ltd., Japan) were used throughout.
Samples were cleaned using oxidising solutions known to remove any non-diamond carbon, leaving the diamond surface terminated with oxygen. Intrinsic undoped diamond epilayers of ca. 600 nm thickness and ca. 2 nm rms average roughness (as determined by atomic force microscopy, AFM) were deposited using PECVD in both ASTeX AX2050 and Seki Technotron Corp. AX5010 plasma reactors using the parameters shown in Table I. Heavily-doped boron layers were deposited in a ‘NIRIM-type’ PECVD reactor using the conditions given in Table II. δ-doped layers had average roughnesses of ca. 2.5 nm (as judged by AFM). Plasmas were extinguished 1 min after the CH₄ flow was shut off and all samples were cooled for 20 mins in 50 Torr, 400 sccm H₂ ambient. Prior to PECVD, all reactors were shut off and all samples were cooled for 20 mins in 50 Torr, 5 x 10⁻⁴ mbar. Ultra low energy SIMS measurements were then performed within the temperature range of 300 to 525 K (in 25 K increments) and further analysis, such as fitting routines, were performed using Originpro software. Measurements were repeated 3 times at each temperature; errors in the measurement are not plotted as they are too small to visualize. Before measurements were performed, samples were put under vacuum and subjected to a dehydration bake of 500 K. Ultra low energy SIMS measurements were performed using an Atomika 4500 Quadrupole SIMS instrument under ultrahigh vacuum (5 x 10⁻¹⁰ mbar) using an oxygen primary ion beam with the following parameters: energy 250 eV; current ca. 30 nA; area ca. 180 μm by 180 μm and normal incidence. Boron concentrations were calibrated using a PECVD boron doped diamond reference sample (ref: D121, ca. 800 nm, nominal concentration 2 x 10²¹ cm⁻³), and further analysis, such as fitting routines, were performed using Originpro software.

### III. RESULTS

#### A. AC Hall effect

AC Hall measurements have been performed to measure the sheet carrier density and mobility of the boron-doped layers. Sample A represents a relatively thick heavily doped diamond and B & C are δ-doped layers. AC Hall measurements reveal all samples to have positive Hall coefficients indicating that holes are the majority carriers (Table III). Assuming similar doping concentrations, the first δ-layer, sample B, shows mobilities in excess of what is expected for thicker heavily boron-doped diamond, as indeed is the case in comparison to sample A (< 3 cm²/V.s) (Fig. 1). Similarly, the sheet carrier density for sample B is sufficiently reduced (in comparison to sample A) to be controllable by a FET gate. PECVD recipe alterations then resulted in sample C, which shows further mobility enhancement to around 13 cm²/V.s, and again a desirable sheet carrier density. The full sets of properties determined are given in Table III.

#### B. Impedance spectroscopy

IS has been used to determine the dielectric properties and activation energies of the channels in order to estimate the boron concentration of the δ-layers. Impedance spectroscopy is used in conjunction with SIMS due to SIMS being inaccurate within the first few nanometres of a sample. IS measurements are performed on samples B & D in air and vacuum from 300 K (room temperature) to 525 K, where D is of the same PECVD recipe as C. Sample temperatures have been intentionally kept below 525 K in order to maintain the oxygen coverage of the layer, which is known to affect the electrical characteristics of boron-doped surface channels. Bode plots, shown in Fig. 2, give the real and imaginary impedance values (Z’ and Z”) of samples B & D in air and vacuum (10⁻³ Torr) from 300 K to 525 K. The high frequency resistance increases seen in the Bode plots are due to the high frequency skin effect from the source and drain contacts. The high cut-off frequency of ca. 10⁷ Hz in samples B and D show them to have little capacitance in both air and vacuum. This suggests a metallic-like nature, which is expected for heavily doped layers. Increasing the temperature of the samples results in the impedance of the samples reducing in a logarithmic relationship, as expected for thermally activated carriers.

### Table I. PECVD parameters for epilayer depositions.

<table>
<thead>
<tr>
<th>Sample</th>
<th>(B/C)ₑₓₑ</th>
<th>CH₄</th>
<th>CH₄</th>
<th>H₂</th>
<th>Power</th>
<th>Pressure</th>
<th>Temp.</th>
<th>Time</th>
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</thead>
<tbody>
<tr>
<td>A</td>
<td>6000</td>
<td>3</td>
<td>1.8</td>
<td>3</td>
<td>95.2</td>
<td>600</td>
<td>80</td>
<td>~800</td>
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<tr>
<td>B</td>
<td>6000</td>
<td>3</td>
<td>1.8</td>
<td>3</td>
<td>95.2</td>
<td>600</td>
<td>80</td>
<td>~800</td>
</tr>
<tr>
<td>C&amp;D</td>
<td>600</td>
<td>0.3</td>
<td>0.18</td>
<td>0.3</td>
<td>99.5</td>
<td>600</td>
<td>80</td>
<td>~800</td>
</tr>
</tbody>
</table>

### Table II. PECVD parameters of heavily boron-doped diamond layers. Sample A&B use high conc. (h) TMB at 1.09% (in H₂), and C&D use low conc. (l) at 0.0977%.

<table>
<thead>
<tr>
<th>Sample</th>
<th>(B/C)ₑₓₑ</th>
<th>CH₄</th>
<th>CH₄</th>
<th>H₂</th>
<th>Power</th>
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<td>3</td>
<td>95.2</td>
<td>600</td>
<td>80</td>
<td>~800</td>
</tr>
<tr>
<td>C&amp;D</td>
<td>600</td>
<td>0.3</td>
<td>0.18</td>
<td>0.3</td>
<td>99.5</td>
<td>600</td>
<td>80</td>
<td>~800</td>
</tr>
</tbody>
</table>
and by applying linear fits to the log of impedance versus reciprocal temperature, the gradient of the slope has been used to determine the activation energy of the conductive channels. Arrhenius plots of the B & D in Fig. 3 reveal B to have an activation energy of 22 meV in vacuum and two activation energies in air of 47 meV below ca. 400 K and 141 meV above 400 K. Sample D exhibits a reduced activation energy in comparison to B of 10 meV in vacuum and a similar dual activation energies in air of 21 meV below 375 K and 85 meV above. The low activation energies of B & D are ascribed to nearly fully activated carriers forming a metallic impurity band, indicating the channel to be heavily-boron doped. In air, the two linear fits could possibly be ascribed to thermally induced changes in the surface barrier of the uncapped channel from the onset of oxidation of the surface in air; however, the observed changes are thermally reversible.

C. Secondary ion mass spectroscopy

Ultra low energy secondary ion mass spectroscopy (ule-SIMS) has been used to investigate the concentrations and distributions of boron in δ-doped samples B & D. Raw SIMS data reveal samples to have peak boron concentrations of $2 \times 10^{19}$ cm$^{-3}$ and $1 \times 10^{21}$ cm$^{-3}$ for B & D, respectively [Figs. 4(a) and 4(b)], located at the surfaces of the samples with comparable concentrations to those estimated in IS. Analysis of the substrate-side interface of the δ-layers shows the raw boron concentration to drop at slow rates of ca. 35±3.5 nm/decade and 9.5±0.1 nm/decade for B & D, respectively. This is unexpected, considering the minimal boron diffusion that occurs in diamond and the PECVD method of CH$_4$ injection employed in this study to deposit δ-layers B & D. In fact, the close-fitting exponential decay of the boron concentration into the substrate [see Figs. 4(a) and 4(b)] indicates it to be representative of a convoluted SIMS response function as a consequence of the common phenomenon of primary ion beam mixing$^{30}$ (i.e., ‘knock-on’ of boron). Therefore, a simple deconvolution algorithm$^{31}$ using smoothed raw data has been used to extract a better approximation of the actual boron profile in samples B & D. Figures 4(c) and 4(d) show the deconvoluted boron concentrations of B & D to have more δ-like profiles with the

![FIG. 1. AC Hall measurements at room temperature (300 K) for samples A, B, and C.](image1)

![FIG. 2. (Color online) IS data for samples B (a),(b) and D (c),(d) in vacuum (a), (c) and air (b),(d) over the temperature range of 300 to 525 K.](image2)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sheet resistance [Ω/square]</th>
<th>Hall coeff. [cm$^2$/V.s]</th>
<th>Sheet Carrier Density [cm$^{-2}$]</th>
<th>Mobility [cm$^2$/V.s]</th>
<th>Carrier type</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>3.32</td>
<td>0.43</td>
<td>$1.94 \times 10^{18}$</td>
<td>1.24</td>
<td>Holes</td>
</tr>
<tr>
<td>B</td>
<td>$7.95 \times 10^4$</td>
<td>77.77</td>
<td>$8.03 \times 10^{12}$</td>
<td>9.78</td>
<td>Holes</td>
</tr>
<tr>
<td>C</td>
<td>$3.82 \times 10^4$</td>
<td>51.05</td>
<td>$1.22 \times 10^{13}$</td>
<td>13.35</td>
<td>Holes</td>
</tr>
</tbody>
</table>
majority of boron segregated into the first two nanometres of the samples. Peak boron concentrations of ca. 8.2×10^{19} \text{cm}^{-3} and 1.4×10^{21} \text{cm}^{-3} and boron concentration gradients of 2.0±0.5 nm/decade and 0.86±0.1 nm/decade are estimated for samples B & D, respectively. A relatively high background boron concentration is recorded for both samples [Figs. 4(a) and 4(b)], which is ascribed to the continual sputtering of boron from the δ-surface at the primary ion beam crater periphery, as well as boron contaminants lingering in the SIMS chamber. This high background noise level also means the accuracy of such analysis is clearly limited.

IV. DISCUSSION

AC Hall measurements show δ-doped samples to have room temperature mobilities in excess of what is expected for thicker heavily boron-doped (111) diamond layers at metallic boron concentrations (>10^{20} \text{cm}^{-3});\textsuperscript{16,32} however, in the absence of direct measurements of boron concentration, the origin of these elevated mobilities are not immediately apparent. It is well known that diamond doped with a lower boron concentration results in carriers with higher mobilities and larger activation energies.\textsuperscript{3,33} This leads to the suggestion that the measured mobilities could actually be ascribed to conductive channels with lower than expected boron doping concentrations and the corresponding higher mobilities. However, the mobility measured in both samples B and D do not correspond to the typical mobility of heavily boron-doped diamond with an activation energy ranging from 20 to 140 meV. Comparing the activation energy of impedance of sample B to literature values\textsuperscript{16,32} of activation energy versus boron concentration, the 22 to 140 meV \(E_a\) measured corresponds to boron concentrations of 10^{19} to 10^{20} \text{cm}^{-3}. Similarly for sample D, the 10 to 85 meV \(E_a\) corresponds to 10^{20} to 10^{21} \text{cm}^{-3} boron concentrations. For thick boron-doped diamond (111) of these boron concentrations the mobilities recorded in the literature\textsuperscript{16,32} are typically 1–2 cm\textsuperscript{2}/V.s, as demonstrated in sample A (Fig. 1). Vice-versa, sample B’s measured mobility of ca. 13 cm\textsuperscript{2}/V.s would correspond to doping concentrations of 10^{17} to 10^{18} \text{cm}^{-3} which would exhibit activation energies in the range of 300 meV. Indeed, SIMS measurements show IS estimated boron concentrations
to correspond well to measured values, and show the PECVD recipe of sample D to significantly improve on the recipe used for sample B; however, estimated boron profile gradients for samples B & D are still in excess of the Debye Length in diamond, $L_D$, suggesting hole delocalisation may not occur effectively in these samples\textsuperscript{14} ($L_D$ in B: $\sim$0.3 nm, D: $\sim$0.7 Å). On the contrary, for such challenging SIMS samples as uncapped diamond $\delta$-layers within the top nanometre of samples, peak displacement and broadening effects could easily be broadening and shrouding steeper-profiled $\delta$-doped layers. Such effects include primary ion beam mixing as already discussed, and additional effects such as pre-equilibrium non-linear sputtering, topography development,\textsuperscript{30} and sample roughness.

When considering the electrical properties of uncapped $\delta$-layers, the atomic surface termination, and thereof the energetic surface barrier of the channel, is important in determining the level of carrier depletion and surface defects. Oxygen terminating the surface of (100) boron-doped diamond is known to create a surface barrier that pins the Fermi level and, if sufficiently large (ca. 1 eV), can completely deplete thin (ca. 1 nm) channels of hole carriers.\textsuperscript{29,35} However, it has been suggested that oxygenated (111) diamond has a low density of surface states and as a result does not have a pinned Fermi level or surface barrier.\textsuperscript{36} The surface barrier created by the UV ozone treatment used here has not been measured directly. However, since the measured sheet carrier densities are close to the expected sheet carrier densities estimated from bulk doped diamond properties and PECVD parameters, this suggests that no surface pinning, that causes significant carrier depleting, is occurring.

When AC Hall measurement observations are combined with the high cut-off frequency (ca. $10^5$ Hz) measured in IS, the nature of the mobility enhancement in the boron-doped layers can be ascribed to the onset of $\delta$-like properties causing a fractional delocalisation of holes out of the $\delta$-layer. While the boron-doped layers display enhanced hole mobilities, further work is required to improve the carrier mobility of the channels. Firstly, the $\delta$-layers grown here are uncapped. It has been suggested that the capping layer of a $\delta$-layer contains the largest portion of delocalised holes\textsuperscript{7} and therefore should account for a large enhancement in the mobility value of the channel. Furthermore, the level of boron doping estimated in sample B ($10^{19}$ to $10^{20}$ cm$^{-3}$) is not sufficient to fill the higher energetic hole states of a 2D gas which have higher mobility,\textsuperscript{7} and for both samples a sufficiently sharp boron concentration gradient has not been measured. Thus, further mobility enhancements should be possible with sharper $\delta$-interfaces, an intrinsic capping layer of diamond on top of the $\delta$-layer and the use of flatter substrates. Finally, further refinements to the PECVD recipe are possible to improve the $\delta$-layer quality.

V. CONCLUDING REMARKS

Uncapped $\delta$-doped layers have been deposited on (111) diamond substrates, wherein a revised PECVD protocol has been employed to aid abrupt $\delta$-doped boron concentration profiles. AC Hall effect and dielectric impedance spectroscopy measurements have been used to determine the electrical characteristics of $\delta$-layers, and enhanced room temperature mobilities (13 cm$^2$/V.s) are reported in comparison to equivalently grown thicker heavily boron-doped (111) diamond. Impedance spectroscopy measurements show the deposited layers to have metallic-like properties with low activation energies corresponding to estimated boron concentrations of approximately $10^{19}$ cm$^{-3}$, as confirmed by SIMS measurements. The resulting boron $\delta$-doped diamond channels are suitable for FET fabrication with desirable sheet carrier densities.

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