The Effect of Substrate Temperature and Growth Rate on the Doping Efficiency of Single Crystal Boron Doped Diamond

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Abstract

The substrate growth temperature dependence of the plasma gas-phase to solid-phase doping efficiency in single crystal, boron doped diamond (BDD) deposition is investigated. Single crystal diamond (SCD) is grown by microwave plasma assisted chemical vapor deposition (MPACVD) on high pressure, high temperature (HPHT) type Ib substrates. Samples are grown at substrate temperatures of 850-950°C for each of five doping concentration levels, to determine the effect of the growth temperature on the doping efficiency and defect morphology. The substrate temperature during growth is shown to have a significant effect on the grown sample defect morphology, and a temperature dependence of the doping efficiency is also shown. The effect of the growth rate on the doping efficiency is discussed, and the ratio of the boron concentration in the gas phase to the flux of carbon incorporated into the solid diamond phase is shown to be a more predictive measure of the resulting boron concentration than the gas phase boron to carbon ratio that is more commonly reported.

Keywords

microwave plasma chemical vapor deposition (MPCVD); boron doped diamond; doping efficiency; defect morphology; infrared spectroscopy.

Introduction

Diamond is an exceptional material, not only for its well-known mechanical properties, but also for electronic properties such as a wide bandgap, high breakdown voltage and high electron and hole mobilities. As an electronic material, diamond has the potential to revolutionize high-power, high-frequency devices, especially when operating in high temperature environments. The vertical architecture

Schottky barrier diode is one such application where diamond would be particularly well suited [1]. The realization of vertical diode structures requires the ability to reliably produce free-standing ($> 300\text{ }\mu\text{m}$) boron doped diamond substrates, with low resistivity via heavy doping ($> 10^{20}\text{ cm}^{-3}$).

One of the obstacles in achieving heavily boron-doped free standing single crystal diamond substrates is that of the doping efficiency. Several groups [1-3] have reported that a lower percentage of boron from the plasma feedgas is incorporated into the grown solid phase diamond as the boron to carbon ratio in the plasma feedgas increases. In our previous work [4] we saw higher doping efficiencies than what was predicted in the work of Achard *et al* [1,2], and we suggested that it might be due to the higher substrate temperatures during growth that we used in our growth processes. To better understand the role of substrate temperature during growth, a series of experiments that varied the growth temperature and boron to carbon feedgas ratio have been performed.

Material and Methods

Boron doped diamond was deposited on 3.6 mm x 3.6 mm type Ib HPHT diamond seeds with nominal thicknesses of 1.4 mm using a microwave plasma-assisted CVD reactor [5]. The growth surfaces were measured by XRD to be less than 1° off of the (100) crystallographic orientation. The reactor operates at 2.45 GHz with a molybdenum substrate holder that is water cooled. In all of the deposition experiments of this work, the reactor pressure of 240 Torr, the methane concentration of 3.75% in the plasma feedgas, and the total flow rate of 400 sccm were held constant. Diborane was used as the boron source in the feedgas. Using this growth regime, three growth temperatures were initially tried, and the resulting grown defect morphologies are shown in **Figure 1**. The sample grown at 1050°C showed a large number of pyramidal hillock type defects and polycrystalline regions. **The formation of polycrystalline regions at higher deposition temperatures may be expected, and has been noted by other groups as well [6].** Soot deposition during boron doped diamond growth is known to increase with increasing microwave power levels [2], and so there were additional concerns that increasing the microwave power to achieve higher temperatures would produce more soot deposition at higher doping levels. As a result, 1050°C was eliminated as a temperature from the experimental design. Samples were therefore grown at either 850°C or 950°C, as measured **at least hourly during deposition** by one-color optical pyrometer. **During the temperature measurement, the handheld pyrometer is focused across many points on the sample surface, and a number**

of readings are taken. The highest repeatable measurement taken while in focus on the sample surface is used as the temperature result. Measurements taken this way show good stability and repeatability, and are not subject to the inconsistencies associated with a fixed pyrometer that can sometimes move or lose focus during a long deposition. It is expected that the effect of taking the measurement partly through the bottom edge of the plasma ball does not contribute significantly to the measured temperature, since measuring the temperature of the holder next to the sample through the plasma ball yields a temperature below the detection threshold. Only the temperature during growth and the diborane concentration were varied in the experimental design, and the other deposition variables were held constant in order to minimize the influence of other factors on the growth. The deposition conditions are given in **Table 1**.

The diamond substrates were acid cleaned prior to being loaded into the reactor, which is pumped down to a base pressure of < 0.1 mTorr. Substrates are hydrogen plasma etched for approximately 20 minutes during the reactor ramp up procedure, during which the reactor pressure is brought up from the point at which the plasma is started at 5 Torr, to the growth pressure of 240 Torr. Deposition times are counted from the point when the reactor reaches 240 Torr and the methane flow is started. Depositions were run for 8 hours, except for the two highest doping concentration runs, which had to be stopped early due to soot deposition on the reactor quartz dome after 6 hours (SND33) and 7.5 hours (SND34). Soot deposition places an upper limit on the possible duration of any heavily boron doped growth run [2], however it is possible to achieve thick films of over 300 μm by doing either lower boron concentration runs or multiple subsequent deposition runs on the same substrate. In this work only single deposition experiments were performed.

Microwave power was varied between 1.5 kW and 2.2 kW to maintain a constant substrate temperature, as given in **Table 1**. The microwave power was adjusted as needed during the runs, and most samples required greater applied microwave power at the beginning of deposition runs to maintain the set point temperature than near the end of the deposition. The microwave power is therefore given as a range in **Table 1**. The as-received type Ib HPHT substrates can vary in thickness by 100 μm or more, and since the same sample holder set was used for all the depositions, the thinner substrates were used for the 850°C experiments, and thicker substrates were used for the 950°C experiments. This was done to minimize the difference in the applied microwave power needed to maintain the required substrate temperatures, and

therefore as similar a plasma chemistry as possible was maintained for the depositions from run to run. It should be noted however, that varying the applied microwave power from as low as 1.5kW to as high as 2.2 kW may have an effect on the microwave power density, and therefore the plasma chemistry. The samples grown at 950°C did require slightly higher applied microwave power, averaging 1.74-1.96 kW, while the samples grown at 850°C averaged 1.58-1.9 kW. These two ranges have significant overlap, and the microwave power used for individual samples is quite similar. As a result, we attribute the differences noted in this work in samples from the two temperature set-point groups to be due to the growth temperature, rather than primarily due to a change in plasma chemistry.

Sample thickness is measured using a Solartron Metrology Linear Encoder (Model DR600) at several locations in the sample center, which is the region exposed for Fourier Transformed Infrared (FTIR) transmission spectroscopy characterization. The thickness at the corners is measured at several spots at each corner and given as an average over all four corners. Growth rates of 1-5 $\mu\text{m/hr}$ at the center and 4-11 $\mu\text{m/hr}$ at the corners were obtained, as given in **Table 1**.

The analysis of the FTIR transmission spectrum focused on the absorption feature at 1290 cm^{-1} , which is in the one phonon absorption range. Above boron concentrations of approximately 10^{18} cm^{-3} , the signal to noise ratio of the contribution due to the boron in the grown film becomes strong enough that an analysis of the 1290 cm^{-1} feature can be performed with an appropriate calibration to determine the boron content of highly doped samples. The FTIR transmission spectrum was measured using a circular aperture with a 1.5 mm radius. For each sample, spectra are first taken for the growth substrate prior to deposition, and then for the grown diamond film on the substrate after deposition. The absorption coefficient α was determined using **equation 1**, where T_{doped} is the transmission spectrum of the sample after growth, $T_{baseline}$ is the transmission spectrum of that sample's undoped HPHT seed prior to deposition, and t is the film thickness, measured in cm. The assumption implicit in this method of calculating the absorption coefficient is that any change in the transmission spectrum is due to the grown diamond film, and therefore any new absorption takes place within the grown thickness.

$$\alpha = -\frac{\ln\left(\frac{T_{doped}}{T_{baseline}}\right)}{t} \quad (1)$$

The absorption coefficient at 1290 cm^{-1} is related to the boron concentration by the relationship in **equation 2**, as given in Davies [7].

$$[\text{B}] (\text{cm}^{-3}) = 2.13 \times 10^{17} \alpha(1290\text{ cm}^{-1}) (\text{units: cm}^{-1}) \quad (2)$$

In a study of the infrared absorption of $3\text{ }\mu\text{m}$ thick homoepitaxial diamond films grown on type Ib (100) substrates, Gheeraert *et al.* [8] confirmed that for boron doped diamond films with $[\text{B}] > 10^{18}\text{ cm}^{-3}$, the relationship of **equation 2** gives boron concentrations very close to SIMS measurements. In a more recent work, Achard *et al.*[2] also found excellent correspondence between SIMS measurements and the relationship of **equation 2** for their freestanding heavily boron doped single crystal diamond samples.

As the heavily doped samples grown in this work all have boron concentrations over 10^{18} cm^{-3} , **equation 2** is expected to give an accurate estimate of the boron content, as has been demonstrated by other groups [2,8]. Four of the samples of this work were also measured by Secondary Ion Mass Spectroscopy (SIMS) by Evans Analytical Group (www.eaglabs.com), and the results of those measurements will be compared to the FTIR estimated concentrations.

Results

Temperature Dependent Defect Morphology

Optical micrographs of the grown sample surfaces are given in **Figure 1** and **Figure 2**. The samples grown at 950°C show a different defect morphology, with virtually none of the unepitaxial crystallite defects that dominate the surfaces of the samples grown at 850°C . The reflection mode optical microscopy images shown in **Figure 1** and **Figure 2**, which are used for comparison of defect morphologies, tend to exaggerate the yellow color of the grown films. For comparison, an as-received HPHT seed and two of the samples of this work are shown in the photograph in **Figure 3**, where the heavily doped sample appears much darker than in the optical micrographs.

One possible mechanism by which the samples grown at 950°C grow smoother, with less unepitaxial crystallite defects than those grown at 850°C , could be related to the increase in the migration of the carbon species across the epitaxial surface during growth. At higher growth temperatures the carbon species may migrate more during growth. For lower simulated deposition temperatures, May *et al.* have shown that

increasing migration events gives smoother films than direct absorption dominated growth processes with fewer migration events [10].

Temperature Dependent Doping Efficiency

To determine if the substrate growth temperature had any significant effect on the plasma gas to solid phase diamond boron doping efficiency, the boron concentration as estimated by FTIR was plotted against the [B]/[C] ratio in the plasma feedgas as shown in **Figure 4**. For the same [B]/[C] ratio in the plasma gas phase, the samples grown at 950°C show a higher level of boron incorporated into the grown films, suggesting that there is a temperature dependency to the doping efficiency. The samples grown in this work also show a higher doping level than samples from the recent literature [2], which are shown in **Figure 4** as red circles for comparison with the data of the current work. Additionally, the boron concentrations for the samples of this work do not seem to show the saturation behavior at high gas phase [B]/[C] ratios seen by other groups [1-3], indicating that perhaps the growth conditions of this work do not result in the decreasing doping efficiency that has been noted elsewhere. A decrease in the doping efficiency may therefore be due to some factor other than the growth chemistry itself, for example the reactor geometry or the feedgas flow path.

Discussion

Temperature Dependence of the Center Growth Rate

The growth rate results given in **Table 1** seem to indicate a temperature dependence to the sample growth rate, with the samples grown at 950°C growing slower in the center regions than the samples grown at 850°C. This is an unusual result, as the growth rate is generally seen to increase with the growth temperature. For example, Hamada *et al* [9] saw for their undoped single crystal diamond that the growth rate increased with temperature, which they attributed to an increase in the apparent lateral growth, which also improved the crystalline quality.

The mechanism by which the 950°C samples in this study grew slower in the center than the 850°C samples must therefore be a different mechanism, but could perhaps be explained with a simple model, which is related to the higher crystalline quality in the center of the 950°C samples. The nucleation of new lattice planes can be the limiting step in smooth epitaxial diamond growth, and with the absence of the unepitaxial crystallites in the center of the 950°C samples, which may provide additional nucleation sites

for the samples grown at 850°C, the samples grown at 950°C grow slower. The decrease in the center growth rate with increasing temperature between the 850°C and 950°C samples may therefore be due to the concomitant decrease in defects, rather than due to the increase in temperature.

Boron to Carbon Flux Ratio More Predictive of Doping Level

The doping efficiencies achieved in this work are higher than those reported by Achard *et al.* [2], as seen in **Figure 4**, which is cause for some concern. The [B]/[C] ratio in the plasma feedgas seems to be a poor predictor of the boron concentration in the resulting diamond. With the same [B]/[C] ratio, samples could have widely varying doping concentrations, differing by as much as a factor of 10. We have shown that the substrate temperature may play a role in the doping efficiency, however the samples of Achard *et al.* were reported to have been grown at 850°C as well, and even if there is a difference in the calibration of the actual value of the temperature measurements between the two groups, the difference in the two doping efficiencies is still likely to be too large for the temperature alone to account for it.

One significant disparity between the samples of this work and the samples of Achard *et al.* are the differences in the growth rates of the samples of these two works. Achard *et al.* report growth rates of 10 µm/hr, however the samples in the present work have growth rates, given in **Table 1**, of only 1-5 µm/hr. We theorize that the rate of boron incorporation into the grown diamond film may not increase at the same rate as the total growth rate, and therefore as the growth rate increases, the doping efficiency will decrease. The relevant ratio for the estimation of boron concentration from the growth parameters could therefore be the ratio of the boron in the plasma feedgas to the flux of carbon into the grown diamond. This ratio can be found by taking the [B]/[C] ratio in the plasma feedgas, and dividing it by the growth rate, thereby normalizing the carbon in the feedgas to the rate at which it is incorporated. **Figure 5** shows the data of **Figure 4** reinterpreted this way, using the growth rates of **Table 1** for the samples of this work, and 10 µm/hr for the samples of Achard *et al.* **Figure 5** shows a much more linear relationship than **Figure 4**, suggesting that the feedgas boron to “incorporated carbon flux” (feedgas carbon multiplied by growth rate) proposed here is a more predictive measure of the boron concentration as measured by FTIR in a grown diamond film than the [B]/[C] gas phase ratio alone.

Comparison of FTIR and SIMS for Boron Concentration Measurement

SIMS measurements of samples SND25, SND26, SND33 and SND 34 were performed by EAG labs, and the results of these measurements are shown in **Figure 6**. The SIMS measurements were all performed in the center of the samples. The SIMS measurements give high boron incorporation levels, of $8.9 \times 10^{18} \text{ cm}^{-3}$ to $5.8 \times 10^{19} \text{ cm}^{-3}$. These concentrations are 5 to 8 times lower than what was estimated by FTIR, and this discrepancy could be due to a number of factors. The FTIR measurement could overestimate the boron concentration due to imperfect transmission through the grown samples. The slightly angled surfaces at the edges of the samples, especially those grown at 950°C, could reflect or refract the incident IR beam away from the detector beam giving a lower IR absorption measurement. The thicker edge regions also extend slightly into the FTIR aperture window, which would further increase the absorption assumed to occur in the thinner center region. The angled edge regions may also have a higher boron incorporation level, since boron is known to preferentially incorporate on (111) facets [11] and the boron concentration has been shown to increase with the misorientation angle from the (100) surface [12]. The transmission would also be attenuated due to the presence of defects, for example the high density of dark colored unepitaxial defects on the 850°C samples. These sources of error in the FTIR measurement could tend to overestimate the boron concentration, and may be the reason for the discrepancy.

In addition to the sources of possible overestimation of the boron concentration by FTIR, the SIMS calibration was performed by the commercial laboratory, and the details of this calibration and the resulting possible error quantification were not made available. However, even if there is some error in the calibration, the SIMS boron concentrations are still comparable relative to each other. The boron concentrations measured by SIMS show the same temperature dependence of the doping efficiency that was seen in the concentrations determined by the FTIR method, with the samples grown at 950°C showing a higher incorporation of boron than those grown at 850°C. For example, the heavily doped sample SND34 grown at 950°C, was measured by SIMS to have 39% more incorporated boron than SND33, which was grown under the same reactor conditions but at 850°C.

The depth profiles of the SIMS measurements are shown in **Figure 7**. The characterized samples all show good dopant uniformity across the depth of the measurement. The SIMS measurements were all taken in the center of the samples, and although the thicknesses reported in **Table 1** are given as an average over the center regions of the samples, there may be some variation in the thickness in this region. The

grown film of sample SND26 was only 7 μm thick at the point of the SIMS measurement, and the boron concentration that is shown measured after this depth is a SIMS artifact due to the high boron concentration in the grown layer.

Conclusions

Heavily boron-doped diamond samples grown with a substrate temperature of 950°C show fewer defects and higher doping efficiency than those grown at 850°C. This is a significant positive result, as it identifies a reactor regime in which reasonably high quality heavily boron doped diamond can be grown.

The gas phase boron to sample carbon flux ratio proposed here, as obtained by dividing the $[\text{B}]/[\text{C}]$ gas phase ratio by the growth rate, was shown to be a more predictive measure of boron content in the grown diamond than the $[\text{B}]/[\text{C}]$ ratio alone. This suggests that the boron incorporation rate does not increase at the same rate as the growth rate, and therefore faster growth rates will result in lower boron concentration levels.

The saturation of the doping level at high boron concentrations seen in other studies was not seen in this work, and is therefore perhaps due to a factor such as the reactor geometry, and not a mechanism of the growth chemistry.

There are likely other factors that affect the boron doping efficiency in addition to the temperature and growth rate dependences identified here. By identifying the factors which determine the boron doping efficiency, greater control over the boron concentration levels in heavily doped diamond films can be achieved.

The ability to produce defect free, heavily doped, thick diamond substrates with well controlled boron concentrations at a controlled growth rate is a necessary technology for the achievement of vertical Schottky Barrier Diodes. The factors affecting the doping efficiency identified here, substrate growth temperature and growth rate, and the identification of a regime in which defects are substantially reduced, are significant results. The results of this work indicate progress toward the goal of controlling the grown boron concentration levels and the optimization of the growth morphology, which will be necessary to enable high quality boron doped diamond layers.

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Figures

Figure 1: Optical micrographs, taken in reflection mode, showing the defect morphologies of samples grown with $[B]/[C] = 666$ ppm in the plasma gas phase at three different substrate growth temperatures: (a) 850 °C, (b) 950 °C, (c) 1050 °C. The scale bar for all three images, in the lower left corner of (a), is 500 μm .

Figure 2: Optical micrographs, taken in reflection mode, showing the defect morphologies of samples grown at 850°C (top row) and 950°C (bottom row). The $[B]/[C]$ ratio in the plasma feedgas for each sample is given in the column headings. For comparison, the morphologies of samples grown at 666 ppm were shown in Figure 1. The scale bar for all images, in the lower left of the sample grown at 850°C and 933 ppm, is 500 μm .

Figure 3: Examples of the appearances of samples of this work, shown left to right: an as received type Ib HPHT seed, sample SND22 and sample SND29.

Figure 4: [B]/[C] ratio in the plasma gas phase vs. boron concentration in the grown diamond sample. This figure illustrates graphically the doping efficiency. Samples grown at 950°C are shown with green pentagrams (stars), samples grown at 850°C are shown with blue diamonds, and for comparison, samples from the recent literature [2] measured by FTIR using the relationship of equation 2 and confirmed by SIMS, are shown as red circles.

Figure 5: The data from Figure 4 is reoriented by dividing the plasma feedgas [B]/[C] ratio by the growth rate. The samples of this work are divided by their individual growth rates, which are given in Table 1, and for comparison the samples of Achard *et al* [2] are included as well, using the 10 $\mu\text{m/hr}$ growth rate given in that reference.

Figure 6: [B]/[C] ratio in the plasma gas phase vs. boron concentration in the grown diamond sample measured by SIMS (Evans Analytical Group, www.eaglabs.com). Samples grown at 950°C are shown with green hexagrams (stars), samples grown at 850°C are shown with blue squares.

Figure 7: Depth profile of SIMS measurements showing uniformity of doping profile through the sample depth. The SIMS artifact of sample SND26 is due to the high boron concentration in the grown layer on top.

Tables

Table 1: Experimental deposition conditions

Sample	Growth temperature [°C]	Microwave power during growth [kW]	B/C gas phase [ppm]	Center growth rate [μm/hr]	Corner growth rate [μm/hr]	Grown thickness at center [μm]
SND24	850	1.6-1.8	666	2.1	5.5	16.6
SND22	950	1.7-1.9	666	2.0	10.6	16.2
SND25	850	1.6-1.9	933	5.0	10.1	39.7
SND26	950	1.8-2.2	933	1.2	8.4	9.3
SND27	850	1.6-1.9	1333	2.2	5.1	17.7
SND28	950	1.7-1.9	1333	1.1	4.4	9.0
SND30	850	1.5-1.8	2000	3.1	6.2	24.4
SND29	950	1.5-1.7	2000	2.1	9.1	16.5
SND33	850	1.6-2.1	3333	2.8	8.5	16.7
SND34	950	2.0-2.1	3333	1.5	8.6	11.4