

## Dopant Incorporation Efficiency in CVD Silicon Carbide Epilayers

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### ABSTRACT

In order to ensure reproducible and reliable SiC semiconductor device characteristics, controlled dopant incorporation must be accomplished. Some of the many factors which greatly influence dopant incorporation are the site-competition effect, SiC(0001) substrate polarity, substrate temperature, and the dopant-source reactor concentration. In this paper, dopant incorporation is considered and compared for various dopants in the context of dopant incorporation efficiency. By using secondary ion mass spectrometry (SIMS), the relative dopant incorporation efficiencies were calculated by dividing the SIMS determined dopant concentration in the resulting epitaxial layer by the intentional gas phase dopant concentration used during the SiC CVD. Specifically, the relative magnitudes of dopant incorporation efficiencies for nitrogen, phosphorus, and boron in 6H-SiC (0001) Si-face epitaxial layers are compared as a function of the site-competition effect and the dopant-source reactor concentrations. This serves as a first approximation for comparison of the relative "doping potencies" of some common dopants used in SiC CVD epitaxial growth.

### INTRODUCTION

Silicon carbide (SiC) is a high temperature semiconductor material currently being pursued for applications in high temperature, high power, and high frequency electronics<sup>1-5</sup>. SiC electronic devices will find applications in aerospace vehicle control, aerospace power conditioning, space communications, as well as terrestrial control systems<sup>6-8</sup>. However, in order to ensure reproducible and reliable SiC semiconductor device characteristics, controlled dopant incorporation must be accomplished. Some of the many factors which significantly influence dopant incorporation in SiC epitaxial layer growth include the site-competition effect, the SiC substrate polarity (i.e. Si-face or C-face of the SiC(0001) ), substrate temperature, and the dopant-source reactor concentration. Site-competition epitaxy is a recently reported dopant incorporation control technique which allows control over dopant incorporation by adjusting the silicon-source/carbon-source flow ratio (Si/C ratio) into the CVD reactor during epitaxial layer growth<sup>9</sup>. For example, on both 6H- and 4H-SiC(0001) Si-face substrates, nitrogen dopant incorporation was found to be proportional to the Si/C ratio whereas phosphorus, boron, and aluminum dopant incorporation was inversely proportional to the Si/C ratio used in the CVD reactor during epitaxial growth<sup>10, 11</sup>. In addition to the site-competition effect, the SiC substrate polarity has also been recently reported to effect dopant incorporation<sup>12, 13</sup>. For example, secondary ion mass spectrometry (SIMS) results from aluminum doping studies indicate that aluminum dopant incorporation on C-face substrates is 50X less efficient compared to Si-face 6H-SiC substrates<sup>13</sup>.

In this paper, dopant incorporation efficiency will be considered and discussed for phosphorus (P) and nitrogen (N) as n-type and boron (B) as p-type dopants in context of the site-competition effect and dopant-source reactor concentrations. Dopant incorporation efficiency is defined here as the ratio of the SIMS determined dopant concentration incorporated into the SiC epitaxial layer divided by the gas phase dopant concentration used during epitaxial growth. Dopant incorporation efficiency, expressed as a percentage, is only a relative measure for comparing the "doping ability" for each dopant-source under similar growth conditions. The ultimate goal is to provide the reader with a first approximation of the various doping abilities in terms of the relative magnitudes of "reactor dopant concentrations" needed to accomplish a desired amount of dopant

incorporation into a growing SiC epitaxial layer, as well as the relative impact of the site-competition effect on the incorporation of selected dopants derived from specific dopant sources.

## EXPERIMENTAL

Commercially available n-type 6H-(0001)SiC Si-face boule-derived wafers<sup>14</sup> were used as substrates for the 6H-SiC epilayers which were grown in an atmospheric pressure CVD system<sup>15, 16</sup>, with a typical growth rate of 3  $\mu\text{m/h}$ . The SiC substrates were precleaned using a standard degreasing solution, followed by immersion in boiling sulfuric acid for 10 minutes, with a final deionized-water rinse and then dried with filtered nitrogen. The cleaned substrates were placed onto a SiC-coated graphite susceptor and then loaded into a water-cooled fused-silica reactor. The samples were heated via the RF-coupled susceptor which was temperature controlled at 1450°C using an optical pyrometer. Silane (3% in  $\text{H}_2$ ) and propane (3% in  $\text{H}_2$ ) were used as the sources for SiC epilayer growth, whereas a 90 sccm flow of ultra-pure hydrogen chloride gas in a 3 sLpm flow of hydrogen was used during a 1350°C in situ etch prior to epilayer growth. All gases were mass flow controlled, including the ultra-pure hydrogen carrier-gas which was purified by using a heated-palladium diffusion cell. The epilayers were doped n-type using either phosphine or nitrogen and p-type using diborane. For comparison of nitrogen and phosphorus dopant incorporation efficiencies, experiments were performed using similar flows of equivalent atomic source-concentrations of phosphorus (2%  $\text{PH}_3$  in  $\text{H}_2$ ) and nitrogen (1%  $\text{N}_2$  in  $\text{H}_2$ ). Similarly, equivalent atomic source-concentrations of boron (100 ppm  $\text{B}_2\text{H}_6$  in  $\text{H}_2$ ) and phosphorus (200 ppm  $\text{PH}_3$  in  $\text{H}_2$ ) were used for dopant incorporation efficiency comparisons. Secondary ion mass spectrometry (SIMS) was performed<sup>17</sup> using a CAMECA IMS-4f double-focussing, magnetic sector ion microanalyzer, using implanted SiC standards. Cesium bombardment was used for determination of boron, phosphorous, and nitrogen atomic concentration profiles by using the detector in a negative secondary ion detection mode to monitor  $\text{P}^-$  and the diatomic species  $\text{B}(\text{+C})^-$  and  $\text{N}(\text{+C})^-$ , respectively.

## RESULTS AND DISCUSSION

For the following discussions, "dopant incorporation efficiency" is defined as the percentage of dopant which was incorporated into the grown epilayer compared to the amount (atomic concentration) which was available in the reactor during the SiC epilayer growth, calculated using equations (1) and (2):

$$[\text{Dopant}]_{\text{ppm}} = [\text{Dopant}]_{\text{cm}^{-3}} / (\rho_{\text{SiC}}) \quad (1)$$

Equation (1) is used to convert concentrations of atoms/ $\text{cm}^3$  into parts-per-million (ppm). With  $[\text{Dopant}]_{\text{ppm}}$  and  $[\text{Dopant}]_{\text{cm}^{-3}}$  as the SIMS determined dopant concentrations in ppm and atoms/ $\text{cm}^3$ , respectively, and  $(\rho_{\text{SiC}})$  as the density of 6H-SiC ( $4.73 \times 10^{22}$  atoms/ $\text{cm}^3$ , converted from 3.217  $\text{g/cm}^3$ ). Then the dopant incorporation efficiency is simply calculated by using:

$$E = ( [\text{Dopant}]_{\text{SiC}} / [\text{Dopant}]_{\text{Reactor}} ) \times 100 \quad (2)$$

Where E is the calculated incorporation efficiency, and  $[\text{Dopant}]_{\text{SiC}}$  is the SIMS determined atomic dopant concentration in the epitaxial layer whereas  $[\text{Dopant}]_{\text{Reactor}}$  is the gas phase atomic concentration of dopant available in the CVD reactor, both expressed in ppm. Note that the incorporation efficiency is multiplied by 100 to provide a more manageable range of values for ease of comparison and therefore expressed in percentages.

As already stated, comparisons of dopant incorporation efficiency must be considered in context of the dominant factors which influence dopant incorporation during SiC epilayer growth. Previous reports document that dopant incorporation can be controlled by appropriately adjusting the silicon-source/carbon-source ratio (Si/C ratio) contained in the growth reactor during epilayer growth, which is more simply referred to as site-competition epitaxy<sup>9,10</sup>. Additionally, this dopant control technique was reported to function independently of the substrate polytype based on similar results of dopant control obtained for 6H, 4H and 3C-SiC epilayers. Therefore, the following results of dopant incorporation efficiency will be considered in the context of the site-competition effect and dopant source concentration in the CVD reactor during epitaxial 6H-SiC(0001) Si-face growth, but equally apply for the 4H polytype.

#### Phosphorus versus Nitrogen Doping Efficiency

For the following series of doping experiments the silane and propane flow into the reactor were maintained constant and only the dopant flow was increased within each series of epilayer doping experiments. The calculated dopant concentration (atomic) intentionally introduced into the reactor is plotted versus the SIMS determined dopant concentration (atomic) incorporated into the epitaxial layer (see Figure 1). The dopant incorporation efficiency is calculated according to equation (1) and (2). For example, referring to the 2.5% incorporation efficiency results for phosphorus shown in Figure 1, the SIMS determined phosphorus (P) concentration is first converted into ppm by dividing the SIMS P concentration ( $P = 4.8 \times 10^{17}$  atoms/cm<sup>3</sup>) by the density of 6H-SiC ( $\rho = 4.73 \times 10^{22}$  atoms/cm<sup>3</sup>). The P doping efficiency (2.5%) was then obtained by dividing this SIMS P concentration (10.1 ppm) by the gas phase concentration of P in the CVD reactor (400 ppm) during epitaxial growth.

For these Si-face epilayers, grown using a reactor concentration of Si = 200 ppm and C = 1050 ppm (Si/C = 0.19), the SIMS determined phosphorus (P) and nitrogen (N) epilayer concentrations increase as the dopant flow was increased for each epilayer growth-experiment in a series. In contrast, as the reactor dopant concentration was increased, the calculated dopant incorporation efficiencies decrease from 3% to 2.2% for P and from < 1.2% to < 0.6% for N. This incorporation efficiency decrease is consistent with a decrease in available substitutional lattice sites on the SiC growth surface, as the reactor dopant concentration is increased for both P and N<sup>18</sup>. The more efficient P incorporation increases from  $2 \times 10^{17}$  cm<sup>-3</sup> to  $7 \times 10^{17}$  cm<sup>-3</sup> compared to the less efficient N incorporation which increases from  $8 \times 10^{16}$  cm<sup>-3</sup> to  $2 \times 10^{17}$  cm<sup>-3</sup> with equivalent increases in reactor dopant concentrations. The more efficient P incorporation compared to N is somewhat unexpected, especially when considering the much larger atomic size of P compared to that of N. Specifically, the non-polar covalent radius for P is 1.10Å compared to the 33% smaller size of the N atom ( $r = 0.74\text{Å}$ )<sup>19</sup>. However, also by using atomic size as a first approximation (i.e. by neglecting chemical bonding arguments), N should substitute for C ( $r = 0.77\text{Å}$ ) whereas P ( $r = 1.10\text{Å}$ ) should substitute for Si ( $r = 1.17\text{Å}$ ) in the SiC lattice, which is consistent with previous reports<sup>20, 21</sup>. This argument leads to the idea that the amount of available Si-lattice sites (Si-sites) are greater than the amount of available C-lattice sites (C-sites) when using a relatively C-rich Si/C ratio (e.g. Si/C = 0.19), resulting in greater P incorporation because of the relatively more

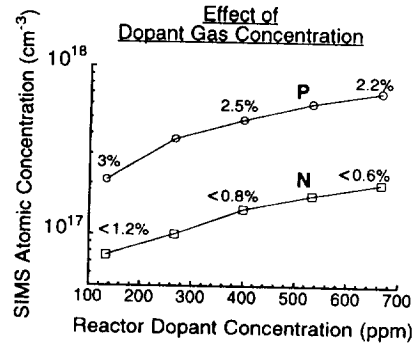


Figure 1 Si-face phosphorus (P) versus nitrogen (N) incorporation efficiencies calculated from using SIMS determined epilayer dopant concentrations divided by the corresponding reactor dopant concentration, while maintaining constant reactor concentrations of Si (200 ppm) and C (1050 ppm), resulting in a constant Si/C ratio = 0.19.

abundant Si-sites compared to the amount of available C-sites. Therefore the site-competition effect (i.e. Si/C ratio effect) must be carefully considered when comparing incorporation efficiencies.

The importance of the site-competition effect can best be illustrated by considering the data presented in Figure 2 in which only the reactor carbon concentration was increased in each successive experiment while the Si (200 ppm), P (200 ppm), and N (200 ppm) were each maintained constant in the CVD growth reactor during separate P and N doping experiments. The intentional reactor carbon concentration is plotted versus the SIMS determined atomic concentration of dopant incorporated during the SiC epilayer growth. The calculated N incorporation efficiency abruptly decreases from 37% to < 1.9% as the reactor carbon concentration increases from 450 ppm to 750 ppm (Figure 2). However, note that the N efficiency is much greater than the P efficiency at C = 450 ppm<sup>18</sup>. Therefore, if the somewhat arbitrary reactor carbon concentration of C = 450 ppm (Si/C = 0.44) had been chosen for experiments of Figure 1 instead of C = 1050 ppm (Si/C = 0.19), then the calculated N efficiencies in Figure 1 would have been greater than the P efficiencies. This variation of dopant incorporation with Si/C ratio is known as the site-competition effect. In this case, the N incorporation decreases as the Si/C ratio decreases because of the increased C competition with N for available C-sites. This teaches that comparisons of P and N dopant incorporation efficiencies are only valid for specific Si/C ratios and cannot be generalized unless the dominant site-competition effect is also considered.

In contrast, the P incorporation efficiency steadily increases from 2.4% to 8.3% as the reactor C concentration is increased. This is also caused by the site-competition effect, but unlike N which is excluded from available C-sites as the reactor C concentration is increased, P incorporation increases because of the increased availability of Si-sites with increased reactor C concentration. In comparing the P doping results shown in Figure 2 with those of Figure 1, note that the calculated P efficiencies of Figure 2 are mostly greater than those of Figure 1 whereas both have similar ranges of SIMS determined dopant incorporation. Therefore, if a phosphorus doped SiC epitaxial layer of  $P = 8 \times 10^{17} \text{ cm}^{-3}$  was desired, the conditions of Figure 1 (C = 1050 ppm; P = 670 ppm) would result in only a 2.2% efficiency compared to a 8.3% efficiency for the growth conditions of Figure 2 (C = 1950 ppm; P = 200 ppm). In other words, a > 3X decreased reactor P concentration could be used to produce an identical P-doped epitaxial layer, which could be important for minimizing potential dopant memory effects, as well as residual contaminant effects, in CVD reactors. These results clearly confirm that N competes with C for C-sites and P competes with Si for Si-sites.

#### Phosphorus versus Boron Doping Efficiency

The dopant incorporation experiments for boron were compared to those for phosphorus using a lower range of reactor dopant concentrations because of the relatively high B incorporation efficiency. For these first series of Si-face epilayer doping experiments, the intentional reactor concentration of Si (200 ppm) and C (1800 ppm) were maintained constant during the growth of each SiC epilayer while only the reactor concentration of dopant was increased. The gas phase

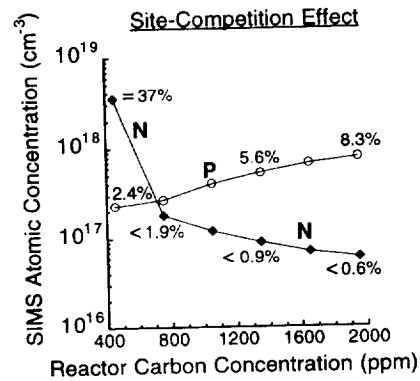


Figure 2 Si-face SIMS determined N and P incorporation efficiencies as a function of decreasing the Si/C ratio (by increasing carbon concentration) using constant reactor concentrations of N (200 ppm), P (200 ppm), and Si (200 ppm) during epitaxial growth.

reactor dopant concentrations (atomic) versus the SIMS determined B and P epilayer concentrations are plotted in Figure 3. The plot with the greater SIMS determined B incorporation (for C = 1800 ppm) does not vary significantly ( $4-5 \times 10^{19} \text{ cm}^{-3}$ ) with increasing reactor B concentration. This first series of B doping experiments have a maximum calculated B incorporation efficiency of approximately 86,000%. In contrast, the corresponding maximum P incorporation is approximately 5000X less efficient (17%), compared to this B efficiency which experienced identical CVD growth conditions. In addition, as the reactor concentration of P is increased, the SIMS determined P incorporation also increases whereas the calculated P incorporation efficiency decreases (from 17% to 13%) as did the previously discussed results using relatively greater P reactor concentrations shown in Figure 1. However, as the reactor concentration of B is increased (C = 1800 ppm), the SIMS determined B incorporation remains relatively constant and therefore the B efficiency decreases with increasing reactor B concentration. It is postulated that this SIMS determined B concentration is approximately at the B solubility limit for these particular CVD growth conditions.

Results for the second series of B doping experiments of Figure 3, using a relatively lower carbon concentration C = 450 ppm (lower B plot in Figure 3), were compared to the first series (upper B plot in Figure 3) which experienced a greater reactor C concentration (C = 1800 ppm). For this second series of experiments, the overall B incorporation decreased from the decrease in the Si/C ratio, consistent with the site-competition effect, where the B mainly competes for the Si-site during the SiC epitaxial growth<sup>11</sup>. Note that as the reactor B concentration was increased (for C = 450 ppm), the B incorporation monotonically increases as does the calculated B incorporation efficiency which increases from 270% to 1700%. This increase in B incorporation with increased reactor B concentration is consistent with the previously discussed N and P results. However, more work is needed in order to explain why the B incorporation should increase so quickly with increased reactor B that the B efficiency actually increases, which is contrary to the efficiency trend observed for N and P.

The greater dopant incorporation efficiency for B for C = 1800 compared to C = 450 in Figure 3, as a result of a relatively greater C-source concentration relative to the Si-source concentration (i.e. a decreased Si/C ratio), serves to illustrate the dominance of the site-competition effect on dopant incorporation

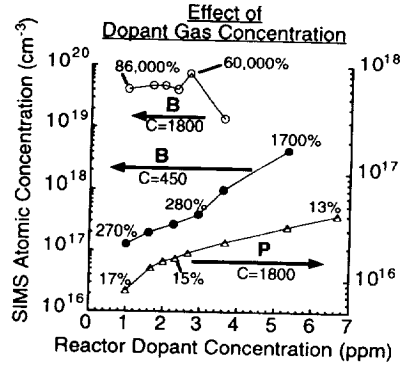


Figure 3 Si-face boron (upper B plot) compared to phosphorus (P) incorporation efficiencies using constant C = 1800 ppm (Si = 200 ppm) and as a function of increasing the reactor dopant concentration. The lower boron plot was performed using C = 450 ppm (Si = 200 ppm) during epilayer growth.

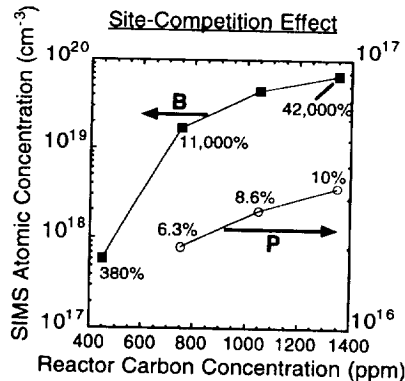


Figure 4 Si-face SIMS determined B compared to P incorporation efficiencies. The B (3.3 ppm) and P(6.6 ppm) reactor concentrations were constant while the Si/C ratio was decreased by increasing the reactor carbon concentration.

efficiency. The dominance of the site-competition effect is further illustrated by the lack of an increase in B incorporation which is normally expected for increased reactor B concentrations (upper B plot in Figure 3). This situation may also be encountered when using uncoated (or insufficiently SiC-coated) graphite susceptors or heated graphite reactor parts in a hydrogen atmosphere, resulting in significantly large concentrations of unintentional C-species in the reactor, produced from the high temperature etching effect of hydrogen on graphite<sup>22</sup>.

The B incorporation efficiency was also compared to the P efficiency as a function of increasing the reactor carbon concentration (i.e. Si/C ratio) to determine the relative magnitude of the site-competition effect, as plotted in Figure 4. For each dopant series, the intentional reactor concentrations of P (6.6 ppm), B (3.3ppm), and Si (200 ppm) were maintained constant during growth of the SiC epilayers. The P incorporation efficiency increases with increasing reactor carbon concentration, with a maximum P incorporation efficiency value of 10% (for C = 1350 ppm and P = 6.6 ppm) as shown in Figure 4. This value is comparable to the P incorporation efficiency of 13% (for C = 1800 ppm and P = 6.6 ppm) from Figure 3, which exhibits a slightly greater P efficiency because of the greater site-competition effect resulting from the relatively greater reactor C concentration used during CVD. As shown in Figure 4, the B incorporation is much greater than the P incorporation despite the decreased reactor concentration of B (3.3 ppm) compared to P (6.6 ppm) used during these separate doping experiments. In addition, the B incorporation efficiency increases from 380% to 42000 %, which represents more than a 100X increase in B efficiency from only a 3X increase (from C = 350 ppm to 1350 ppm) in the reactor C concentration. The much greater B dopant incorporation efficiency compared to the P efficiency, for both constant (Figure 3) and varied (Figure 4) Si/C ratios, can be partially rationalized by considering the relatively small atomic size of the B atom (radius = 0.82Å) compared to that of P (r = 1.10Å) allowing a relatively greater ability of B to substitute for Si (r = 1.17Å) into the Si-site of the growing SiC epitaxial layer.<sup>19</sup>

## CONCLUSION

The dopant incorporation efficiency, defined as the ratio of the resulting epitaxial layer dopant concentration divided by the gas phase dopant concentration, was used as a relative measure for comparison of doping efficiencies for phosphine (P), nitrogen (N), and diborane (B) as dopants that are typically used during the CVD of SiC epitaxial layers. Some of the factors that were discussed, and which influence dopant incorporation efficiency in the CVD growth of SiC epitaxial layers, include the site-competition effect (Si/C ratio) and the dopant source concentration in the reactor during CVD.

These results indicate that dopant incorporation efficiency for the 6H-SiC(0001) Si-face epilayers are greatly dependent upon the site-competition effect (i.e. C-source concentration relative to the Si-source concentration in the reactor during SiC epitaxial growth). The P and B dopant incorporation efficiencies increase whereas N efficiencies decrease with an increasing reactor carbon concentration (i.e. decreasing the Si/C ratio). This is explained by considering that both P and B occupy the Si-sites and that the amount of available Si-sites increase as the Si/C ratio is decreased by increasing the reactor C concentration. Similarly, the N incorporation efficiencies decrease as the reactor C concentration is increased, and caused by the increase in competition from C with the N for available C-sites.

For example, a 3X increase in the reactor carbon concentration (450 ppm to 1350 ppm) resulted in an approximate 100X increase in B efficiency (Figure 4), a 40X decrease in N efficiency (Figure 2), and a 2X increase in P efficiency (Figure 2). This notable change in doping efficiency with a changing Si/C ratio during epitaxial growth, known as the site-competition effect, greatly influences dopant incorporation efficiency and must therefore be considered in order to obtain valid interpretations of dopant incorporation in the growth of SiC CVD epitaxial layers.

For constant Si-source and C-source reactor concentration ratios, as each respective dopant concentration into the reactor was increased, the dopant incorporation efficiencies decrease for N and P, but increase for B. This was partially rationalized by considering the relatively small atomic size of the B atom (radius = 0.82Å) compared to that of P (r = 1.10Å) allowing a relatively greater ability of B to substitute for Si (r = 1.17Å) into the Si-site of the growing SiC epitaxial

layer.<sup>19</sup> However, more work is needed to fully understand the reasons for these incorporation efficiency differences.

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<sup>18</sup>NOTE (*It should be noted, however, that this N incorporation efficiency represents an upper bound because of the potentially significant atmospheric-N background contributions for these SIMS determined epilayer concentrations of N in the < 1-2x10<sup>17</sup> cm<sup>-3</sup> range*).

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