4H-silicon carbide (4H-SiC) is a promising wide bandgap material for high power electronic devices, owing to its unique thermal and electrical properties such as high breakdown field, high thermal conductivity, and high electron saturation velocity.1,2 Chemical vapor deposition (CVD) is a well-developed technique adopted in semiconductor industry to produce high purity and high quality thin films.1 Homoepitaxial growth of SiC via the CVD technique is one of the key steps in the fabrication of high performance SiC devices. Currently, chloride-based CVD has attracted much interest to potentially replace the traditional silane based CVD for SiC epitaxial growth to reduce the Si droplet formation at high growth rates, because of the higher bonding energy of Si-Cl (400 kJ/mol) than Si-Si (226 kJ/mol).4 Dichlorosilane (SiH2Cl2, DCS) is a gas (boiling point: 8.2°C at room temperature). It has been demonstrated that using DCS and propane (C3H8) as the precursors, high crystalline quality 8° off-axis SiC epilayers were achieved at a high growth rate (up to 100 μm/h).5,6 At present, the standard off-axis angle of commercially available SiC substrates is lowered to 4° to reduce the material loss during substrate preparation from the crystal boules. Step-bunching is a typical surface feature of the epilayers grown on 4° off-axis substrates, resulting in increase in surface roughness.7 Morphological defects, specifically the triangular defects and inverted pyramids, are prone to be generated in epigrowth on low off-axis angle substrates.8 Hence, determination of optimized growth conditions to produce SiC epilayers of low defect density with good surface morphology requires a systematic study when evaluating a new precursor system.

It is well known that basal plane dislocations (BPDs) are one kind of performance limiting defects in 4H-SiC bipolar devices. Most of the BPDs in the substrate convert into relatively benign threading edge dislocations (TEDs) during the epitaxial growth.9,10 The rest of the BPDs propagate into the epilayer. Under device current stress, the BPDs in the active layer act as nucleation sites of Shockley staking faults.11,12 The rest of the faults causing increase of forward voltage drift.11,12 This aspect has been reported that the BPD to TED conversion ratio is higher in the case of growth on 4° substrates,13,14 probably related to the interaction between macro-steps and BPDs.9 Also the conversion of a BPD to TED is energetically favorable as discussed in detail in a reference.15 The ratio of elastic energy per unit growth length for a BPD (W_{BPD}) to that for a TED (W_{TED}) can be derived from the references16,17 and written as

\[
\frac{W_{BPD}}{W_{TED}} = \frac{E_{BPD}}{E_{TED} \tan(\beta)} \tag{1}
\]

where \(E_{BPD}\) and \(E_{TED}\) are the elastic energies per unit line length for a BPD and a TED respectively, and \(\beta\) is the substrate off-axis angle (°). Since \(E_{BPD}\) is very close to \(E_{TED}\),16 equation 1 leads to \(W_{BPD} \gg W_{TED}\) when \(\beta \approx 1\), i.e., \(W_{BPD} \gg W_{TED}\). It is energetically favorable for a BPD to convert to a TED during epitaxy. And the smaller the substrate off-axis angle (β), the larger the \(\frac{W_{BPD}}{W_{TED}}\) ratio is. Thus the BPD to TED conversion is greatly enhanced by the reduction of substrate off-axis angle.

It is also demonstrated that the BPD to TED conversion can be enhanced by growth interruptions,18,19 substrate KOH etching,20,22 or epitaxial polishing (CMP) and ready for epigrowth. Then the wafer was diced into 8 mm × 8 mm pieces which were then used as substrates for epitaxial growth in this study. The epitaxial growth was performed in a home-build hot-wall chimney CVD reactor which is...
The growth rate for C/Si ratio increases with C/Si for C/Si < 1, and then reaches saturation for C/Si > 1. This indicates a conversion of the growth regime from C-supply limited (for C/Si < 1) to Si-supply limited growth (for C/Si > 1). This trend is quite common as has been observed in SiC growth using silane or other chloride-based silicon precursors.

Results and Discussion

Growth rate and doping concentration.— The growth rate for the epilayers was investigated at various C/Si ratios (with a constant DCS flow rate of 4.5 sccm). Addition of N₂ does not have an observed influence on the growth rates. As shown in Fig. 2, the growth rate increases with C/Si ratio for C/Si < 1, and then reaches saturation for C/Si > 1. This indicates a conversion of the growth regime from C-supply limited (for C/Si < 1) to Si-supply limited growth (for C/Si > 1). This trend is quite common as has been observed in SiC growth using silane or other chloride-based silicon precursors.

The unintentional doping concentration at various C/Si ratios is shown in Fig. 3. N-type doping is achieved for C/Si < 1.3 and P-type for C/Si > 1.8. This doping dependency behavior is in accordance with the site competition mechanism which was also reported in silane and other chloride-based epitaxial growths. The N atoms replace C atoms in the SiC lattice; thus N incorporation is suppressed by increasing the C/Si ratio. The N incorporation decreases more efficiently at higher C/Si ratios (1.1–1.3). For C/Si ratio above 1.3, the donor concentration is lowered down to the level of residual acceptor impurities. Epilayers grown at C/Si = 1.5 have a doping concentration < 5 × 10¹³ cm⁻³ which is the limit of our C-V mercury probe measurement. High resistivity or semi-insulating epilayers are achieved through a compensation scheme that controls the formation of Si-related vacancies through defect competition. Fig. 3 also shows that the net p-type doping concentration begins to saturate when C/Si ratio reaches 2.2, indicating that the doping type is dominated by nitrogen containing species which are released from the sample holder, hot-wall and insulating material.

The n-type doping concentration increases with the N₂ flow as shown in Fig. 4. It is noted that the N ≡ N bond in N₂ is extremely strong. At the growth temperature (1600°C) N₂ conversion to N-containing species in gas phase is very low. Therefore the kinetic mechanism of surface reaction is important, in which N₂ dissociates and reacts with Si-sites on the surface. Theoretically, the desired highly doped (up to 10¹⁶ cm⁻³) epilayers could be achieved at any C/Si ratio with the addition of sufficient N₂ flow. It is therefore necessary to determine in which condition the epitaxial growth is the most efficient and the epilayers have the highest quality in terms of morphology and defects. This will be discussed in the following sections.
Surface roughness and step-bunching.— The surface morphology of the above epilayers was studied by AFM. The RMS roughness was measured in 20 μm × 20 μm areas and the values are plotted in Fig. 5. As for the unintentionally doped epilayers, the RMS value decreases with increasing C/Si ratio from 0.4 to 1.3, and the value has no significant change (RMS = 1.7–2.0 nm) for C/Si = 1.3–2.2. At C/Si = 2.6, the RMS value reduces to 1.35 nm. Most of the epilayers have obvious step-bunching (Fig. 6b–6e) that is usually observed in low off axis epitaxy. The only exception is the epilayer grown at C/Si = 0.4, which shows a dramatically wavy surface (Fig. 6a) instead of a surface with clear macro-steps. This implies a different growth or step-bunching behavior (Fig. 6b–6d), uniform step-bunching with the macro-step height 4–8 nm is observed on the 4° epilayers grown, and the spaces between macro-steps are quite uniform (0.2–0.3 μm). At C/Si = 2.6 (Fig. 6e), the macro-step height reduces to 1.0–1.5 nm. It is also found that addition of N2 only causes slight degradation in surface morphology. Fig. 5 shows that the RMS values slightly increase by ∼0.5 nm while the N2 flow rate increases from 0 to 15 sccm (the doping concentration increases by 2–4 orders of magnitudes). Considering the above results, it is preferable that the highly n-type doped epilayers are grown at a higher C/Si ratio (>1.3) by simultaneously adding N2 during the growth. However, defect generation and BPD conversion should be considered when determining the most suitable C/Si ratios for epigrowth, which will be discussed in the following sections.

The trend exhibited by the surface roughness RMS value versus C/Si ratio indicates that in epitaxial growth using DCS, step-bunching is enhanced under a Si rich condition. This is opposite to most of the results observed in epigrowth using silane as the precursor, in which step-bunching increased under a C rich condition. However, Ishida et al. also reported that in growth using silane giant step-bunching occurs during epigrowth at very low or high C/Si ratios and they proposed that this is due to the generation of Si or C clusters on the terraces. The enhancement of step-bunching under the Si rich condition was also found by Yazdanfar et al. in the growth using SiH4 + C2H4 + HCl precursor system. Ishida et al. suggested that the optimization of in-situ H2 etching and/or low Cl/Si ratio (which is 2 in DCS) during the growth. The optimization of in-situ H2 etching is suggested by reducing the C3H8 flow rate (or using pure H2), adding HCl, and/or reducing the etching temperature. Addition of HCl to obtain Cl/Si ratio of 3–5 during the growth was found to be an optimum solution for growth in chloride-CVD. Reduction of the growth temperature may also help to moderate/eliminate the step-bunching, though the generation of triangular defects and ingrown stacking faults should be taken into account in low temperature growth conditions.
Morphological defects.—The morphological defects (Fig. 7) are different on epilayers grown at different C/Si ratios. Adding N₂ does not show a clear influence on these morphological defects. The carrot defects (Fig. 7a) and shallow growth pits (5–50 μm dia. Fig. 7b) are mainly observed on the as-grown epilayers grown at C/Si ≤ 0.7 with total densities 5–50 cm⁻². At a C/Si ratio of 1.1, inverted pyramids (Fig. 7c) and triangular defects (Fig. 7d) are frequently observed on the epilayers with total densities <50 cm⁻². It is believed that the Si droplets contribute to the formation of morphological defects in SiC epilayers. Results in this paper imply that Si droplets could still be formed in the Si rich condition using DCS precursor. It is known from a simulation study that in epigrowth using DCS, elemental Si is still generated from decomposition of DCS and its partial pressure is low enough to avoid Si droplet formation only at a very low working pressure (~40 mbar). The reason for Si droplet formation in growth using DCS is most likely due to the low Cl/Si ratio. Addition of HCl into DCS gas system (e.g., Cl/Si = 3-5) during the growth is suggested to be a solution to completely eliminate Si droplet formation even in a Si rich condition.

For C/Si = 1.3–2.6, the epilayers are almost free of the above morphological defects (Fig. 7), except occasionally have some shallow growth pits (similar to amphitheater depressions observed by Burk et al.) in the density <20 cm⁻². With KOH etching of the epilayers, these shallow pits are not associated with any dislocations and believed to be relatively benign for device performance compared with the other morphological defects (e.g., triangular defects). For C/Si ≥ 3.0, very large triangular defects (Fig. 7e) identified as 3C inclusion by Raman spectra are observed. A 3C inclusion can cover fairly large area (e.g., >30 mm² in our study) of the epilayer at C/Si = 3.5.

Basal plane dislocations (BPDs).—After the above characterizations, the epilayers were etched in molten KOH to delineate the dislocations on the epilayer surfaces. Fig. 8 shows the BPD density at various C/Si ratios for unintentionally doped epilayers. The BPD density reduces rapidly with increasing C/Si ratio from 0.4 to 1.1, and then does not show significant change over C/Si = 1.1–2.6. The observed lowest BPD density of 0–5.6 cm⁻² has been achieved in a wide C/Si window. This trend is different from what was reported by Kallinger et al. in the growth using silane precursor. They found that the minimum BPD densities of <15 cm⁻² were achieved for C/Si~1. For lower and higher C/Si ratio, the BPD densities increased. In our study, all of the epilayers have a similar thickness of 6 μm. The change of the BPD density with C/Si ratio could be interpreted by the influence of growth rate. Canino et al. reported that in epigrowth using trichlorosilane precursor the BPD to TED conversion ratio was enhanced by increasing the growth rate. A similar phenomenon may take place in the epigrowth using DCS precursor. For a C/Si ratio of 0.4 the growth rate is low (Fig. 2), resulting in low BPD to TED conversion ratio and thus exhibits a high BPD density (Fig. 8). While the growth rate is higher and almost constant for C/Si ratios of 1.1–2.6 (Fig. 2), steadily low BPD densities are achieved (Fig. 8) because of efficient BPD conversion.

It is also noted that in a slightly C rich condition (C/Si = 1.1–1.5), while the addition of N₂ will achieve n-type doping concentrations up to ~2 × 10¹⁷ cm⁻³, there is no obvious change in BPD densities (Fig. 9). The BPD density in the substrates was found to be ~5000 cm⁻². Hence the BPD to TED conversion ratios greater than 99.8% within 6 μm thick epilayers are achieved for C/Si = 1.1–1.5 regardless of N₂ addition. This is different from what was reported in 8° epitaxial growth where N₂ inhibits the BPD conversion. The

Figure 7. Microscope images of morphological defects on epilayers grown at different C/Si ratios. (a) Carrot defect. (b) Shallow growth pit. (c) Inverted pyramid. (d) Triangular defect. (e) 3C inclusion.

Figure 8. BPD densities at various C/Si ratios for unintentionally doped epilayers.

Figure 9. BPD density vs. doping concentration for epilayers grown for C/Si = 1.1–1.5. (The sample at ~1E13 cm⁻³ is semi-insulating.) Results show that N-doping does not change the BPD density in this C/Si ratio range.
high BPD conversion ratio in our study is among the best reported up to now.13,14,27,37 It should be noted that the previously reported results on 4H epitaxy were obtained in low doped (or unintentionally doped) epilayers, while in this study, this high conversion ratio can be directly achieved in higher n doped epilayers. Therefore the BPD faulting can be effectively suppressed, being of great benefit to SiC bipolar power devices.

In-grown stacking faults (IGSFs).— Generation of IGSFs was also studied in the above conditions. After molten KOH etching of the epilayers, the IGSFs exhibit as two shell-like etch pits (like BPDs) at the down-step side, one is along step-flow direction, another is inclined (as shown in Fig. 10 inset). These two shell-like etch pits may be connected with a shallow groove line.15 These stacking faults are Shockley-type faults with 8H stacking sequence.40 SiC Schottky barrier diodes containing these IGSFs tend to show higher leakage current and reduced breakdown voltage.41

Figure 10 shows the IGSF density at various C/Si ratios. No evident influence of N2 addition on the IGSF generation is found. An IGSF density <20 cm$^{-2}$ was achieved over C/Si = 1.1–1.8. Significant increase of IGSF density is observed at low (≤0.7) or high (≥2.2) C/Si ratios. Abadier et al. postulated that generation of IGSF is correlated with 2D nucleation of adsorbed species on the growing surface at the initial growth condition.40 Data in Fig. 10 imply that either excessive Si or C containing species can enhance the adsorption of unreacted species on the surface contributing to the nucleation of IGSFs. This process is even more severe in the C excess condition.

To reduce IGSF densities at low or high C/Si ratios, elevation of N2 gas. The epilayer morphology and defects are primarily determined by the C/Si ratio. High quality epilayers without morphological defects are obtained at C/Si in the range of 1.3–1.8, even with the addition of N2 for achieving higher n-type doping concentrations. A BPD to TED conversion ratio greater than 99.8% and IGSF density of 0–20 cm$^{-2}$ can be achieved in this C/Si ratio range even in high n doped epilayers.

Conclusions

The characteristics of 4H SiC epilayers grown using dichlorosilane (DCS) as the Si precursor are summarized in Fig. 11. DCS is a promising chlorine based Si-precursor to achieve high quality epilayers with low defect densities. A wide range of doping concentration can be well controlled by the C/Si ratio and simultaneous addition of N2 gas. The epilayer morphology and defects are primarily determined by the C/Si ratio. High quality epilayers without morphological defects are obtained at C/Si in the range of 1.3–1.8, even with the addition of N2 for achieving higher n-type doping concentrations. A BPD to TED conversion ratio greater than 99.8% and IGSF density of 0–20 cm$^{-2}$ can be achieved in this C/Si ratio range even in high n doped epilayers.

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Figure 10. IGSF densities at various C/Si ratios for unintentionally doped epilayers. Inset shows an IGSF after KOH etching of the epilayer.

Figure 11. Chart summary of 4H SiC epitaxial growth using DCS and C$_2$H$_6$ as precursors.
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