

Article

High-Quality 4H-SiC Homogeneous Epitaxy via Homemade Horizontal Hot-Wall Reactor

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Abstract: In this paper, using a self-developed silicon carbide epitaxial reactor, we obtained high-quality 6-inch epitaxial wafers with doping concentration uniformity less than 2%, thickness uniformity less than 1% and roughness less than 0.2 nm on domestic substrates, which meets the application requirements of high-quality Schottky Barrier Diode (SBD) and Metal–Oxide–Semiconductor Field-Effect Transistor (MOSFET) devices. We found that increasing the carrier gas flow rate can minimize source gas depletion and optimize the doping uniformity of the 6-inch epitaxial wafer from over 5% to less than 2%. Moreover, reducing the C/Si ratio significantly can suppress the “two-dimensional nucleation growth mode” and improve the wafer surface roughness Ra from 1.82 nm to 0.16 nm.

Keywords: silicon carbide homoepitaxy; nitrogen doping; horizontal hot-wall reactor



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1. Introduction

Silicon carbide (SiC), as a representative of the third generation of wide-bandgap semiconductor materials, possesses high critical breakdown field strength, excellent thermal conductivity, rapid electron saturation drift speed, wide bandgap width, and strong radiation resistance [1,2]. These characteristics significantly enhance the energy processing capacity of power devices, enabling them to meet the demanding requirements of next-generation power electronic equipment operating under harsh conditions such as higher power outputs, reduced size and volume constraints, elevated temperatures and radiation levels [3,4]. Moreover, SiC offers advantages including size reduction, minimized power loss and decreased cooling demands. Consequently, it has brought about revolutionary advancements in various fields such as new energy vehicles, rail transit systems and smart grids [5]. Therefore, silicon carbide semiconductor materials have gained international recognition as the premier electronic materials that will lead the development of power electronics for at least the next 50 years [6].

With the rapid expansion of the new energy vehicle market, silicon carbide power devices have become highly anticipated [1–6]. Silicon carbide power devices are fabricated on the 4H-SiC epitaxial layer, making silicon carbide epitaxial wafers an indispensable foundational material for these devices [7–10]. The quality of the silicon carbide homogeneous epitaxial layer plays a pivotal role in determining the yield of silicon carbide power devices. Therefore, it is imperative to obtain high consistency and low roughness in silicon carbide epitaxial wafers to ensure the production of high-quality MOSFET devices. Currently, high-quality 4H-SiC epitaxial growth mainly adopts three forms of reactors: horizontal hot-wall, planetary warm-wall, and vertical reactors [11–14]. The epilayers prepared by the horizontal hot-wall reactor achieved good consistency under a uniform temperature field and unique flow field, which has been widely recognized and applied [14].

In this paper, based on the self-developed horizontal hot-wall reactor, we achieved 4H-SiC epitaxy doping uniformity (Doping-U) within 2% and thickness uniformity (Thickness-U) within 1% by exploring and optimizing process parameters such as the carrier gas flow rate and C/Si ratio (twice the flow rate of C_2H_4 divided by the flow rate of TCS). Additionally, the roughness Ra of the domestic epitaxial wafer was reduced to less than 0.2 nm, meeting the quality requirements for high-specification MOSFET devices.

2. Experiments

A homemade horizontal hot-wall epitaxial reactor was utilized for the growth of homogeneous epitaxial layers on domestic 6-inch Si-face 4° off-axis n+ 4H-SiC substrates. The schematic diagram of the reactor is described in Figure 1. Trichlorohydrosilicon (TCS), ethylene (C_2H_4), and nitrogen (N_2) were carried by hydrogen (H_2) carrier gas through three gas-flow tunnels into the reaction chamber, respectively. The 4H-SiC wafer was placed at the center of the gas-foiled rotational susceptor. The substrate was spun with the susceptor during the growth process. The exhaust vent was connected to a butterfly valve downstream of the chamber to control the reaction chamber pressure. During epitaxial growth, the TCS served as the silicon source while C_2H_4 acted as the carbon source. And nitrogen was used as the n-type doping source. The typical process temperature ranged from 1600 to 1650 $^\circ C$, with a variable H_2 carrier gas flow rate of 100–140 slm (standard liter per minute) [15–17], while maintaining a process pressure of 100 mbar. In addition, the 4H-SiC wafer continuously rotated at a low speed along to improve the consistency of growth conditions throughout its surface. The target values for the epitaxial wafer specification were as follows: the average doping concentration and thickness were $8 \times 10^{15} \text{ cm}^{-3}$ and 11 microns, respectively. In addition, different TCS flow rates and hydrogen flow rates were used during epitaxial growth in order to investigate the impact of the C/Si ratio, carrier gas on surface roughness and uniformity of the epitaxial layer.

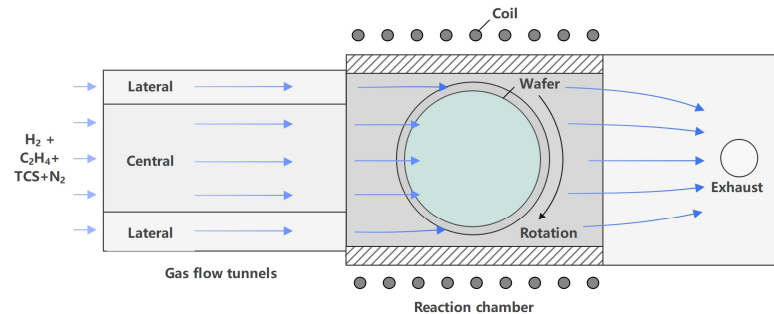


Figure 1. The structure diagram of the SiC epitaxial reactor.

The drift layer thickness trends of 6-inch 4H-SiC epitaxial wafers were characterized using Fourier transform infrared spectroscopy (FTIR iS10, Thermo Fisher Scientific Inc., Waltham, MA, USA), which calculated the thickness of the SiC epi-layer by utilizing the interference effect of difference in the infrared refractive index due to different doping concentrations between the SiC epitaxial layer and substrate. The formula for calculating the epitaxial layer thickness on the substrate is as follows [18,19]:

$$d = \frac{M}{2\sqrt{n^2 - \sin^2\theta}} \cdot \frac{1}{1/\lambda_2 - 1/\lambda_1} \quad (1)$$

where d is the epitaxial thickness (μm); $1/\lambda_2$ and $1/\lambda_1$ are the wavenumbers; M is the number of wave crests between $1/\lambda_2$ and $1/\lambda_1$; n is the refractive index of 4H SiC epitaxy; θ is the incident angle of infrared ray. The doping concentrations of the epi-layer were measured by using a non-contact C-V system (FAaST 230 C-V system, Semilab Semiconductor Physics Laboratory Co. Ltd., Budapest, Hungary). The non-contact C-V measurement of dopant concentration uses corona charge biasing, ΔQ_C , and monitoring of surface voltage, ΔV . Differential capacitance ($C = \Delta Q_C / \Delta V$) was calculated and sequential charging-measuring in

depletion gave the non-contact Schottky barrier C-V, C-Q, and V-Q characteristics [20–27]. These characteristics enable extraction of electrical parameters using well-established procedures, for instance, the standard $1/C^2$ vs. V method gives the dopant concentration, N_D . The calculation formula is shown below [23,27]:

$$N_D = -\frac{2dV}{d(1/C^2) \cdot q\epsilon\epsilon_0} \quad (2)$$

For the calculation of N_D , ϵ is a dielectric constant, ϵ_0 is the permittivity of free space and q is the elemental charge [23]. In order to ensure the accuracy of the testing results, the testing reliability is verified through multiple tests using standard wafers after the instrument calibration is completed. The same point test deviation of the FTIR and the non-contact C-V system on the calibration wafer are less than 0.1% and 0.5%, respectively. As depicted in Figure 2, the test points' locations for doping concentration and thickness analysis are identical, which included selecting twenty-five test points on the wafer surface excluding a five-millimeter edge region. Each test point is equally spaced along two mutually perpendicular diameters: points one and thirteen represent edge points; points two and twelve denote sub-edge points, while point seven is positioned at the center of the wafer. The overall thickness, doping concentration, and uniformity of the whole epitaxial wafer were calculated by using the ratio of standard deviation to the average value of these twenty-five test points in Figure 2. Atomic force microscope measurements using the Bruker Dimension Icon instrument determined wafer surface roughness, whereas defects within the epitaxial layers were identified utilizing the surface inspection system (Candela8520, KLA Corporation, Milpitas, CA, USA) [28–30].

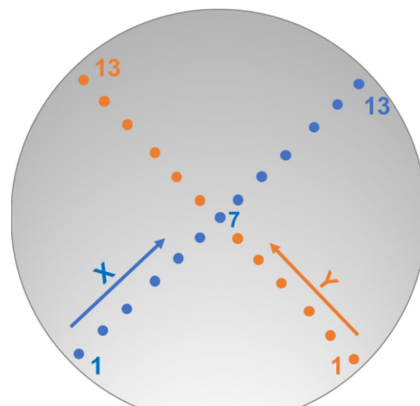


Figure 2. The distribution of test points on the wafer surface.

3. Results and Discussion

3.1. The Impact of Carrier Gas Flow Rate on Doping Uniformity of Epitaxial Layers

The doping concentration and thickness distribution of the SiC epitaxial wafers are presented in Figure 3 for H_2 carrier gas flow rates of 110 slm and 130 slm, respectively, while maintaining a C/Si ratio of 0.85. Based on the data shown in Figure 3, it is apparent that the doping concentration curve on the epitaxial wafer exhibits a classical “W” shape, which is primarily influenced by the different along-track depletion rate of the two source gases and independently controlled dopant (N_2) flow rate in three gas-flow tunnels within the horizontal hot-wall epitaxial reactor (Figure 1) [17,31–35]. Generally, the carbon source exhibits a higher “along-track depletion” rate compared to the silicon source, due to its decomposition and deposition at lower temperatures [36]. When the wafer rotates, it results in a lower actual C/Si ratio at the wafer center compared to its edge. In case there is no additional independent doping source present on the wafer edge within this 6-inch range, a gradual increase in doping concentration can be observed from edge to center on the wafer surface, represented by curve “A” in terms of doping concentration distribution. The inlet area of the epitaxial reactor used in this experiment is divided

into three sections, allowing for independent adjustment of doping concentration at the edge and center of the wafer (by regulating nitrogen flow separately). As a result, the doping concentration on the wafer surface can be distributed in a typical “W” shape along the diameter direction. When the carrier gas flow rate is set at 110 slm (as depicted in Figure 3a), the doping concentration distribution across different points on the wafer is dispersed, and the difference between the center point and the sub-edge point of the wafer is approximately $1.3 \times 10^{15}/\text{cm}^3$, resulting in poor uniformity of 5.2%, which falls below the current international mainstream standards (typically ranging from 3% to 4%). It is somewhat surprising that when increasing the carrier gas flow rate to 130 slm, there is a significant improvement observed in terms of doping concentration uniformity on the epitaxial wafer with an enhanced value of 1.96% (as depicted in Figure 3b), which not only surpasses previous results but also exceeds current mainstream levels. On the other hand, as shown in Figure 3b,c, when the H_2 flow rate increased from 110 slm to 130 slm, the epitaxial layer at the center of the wafer became significantly thicker compared to the edge, and the uniformity of the thickness of the epi-wafer improved from 1.37% to 0.65%. We also found that when the carrier gas flow rate increased from 110 slm to 130 slm, the average thickness of the epitaxial layer decreased from $10.86 \mu\text{m}$ to $10.62 \mu\text{m}$, and the thickness at the center of the wafer reduced more than that close to the edge, which indicated that the growth rate of the epi-layer slowed down as the carrier gas flow increased.

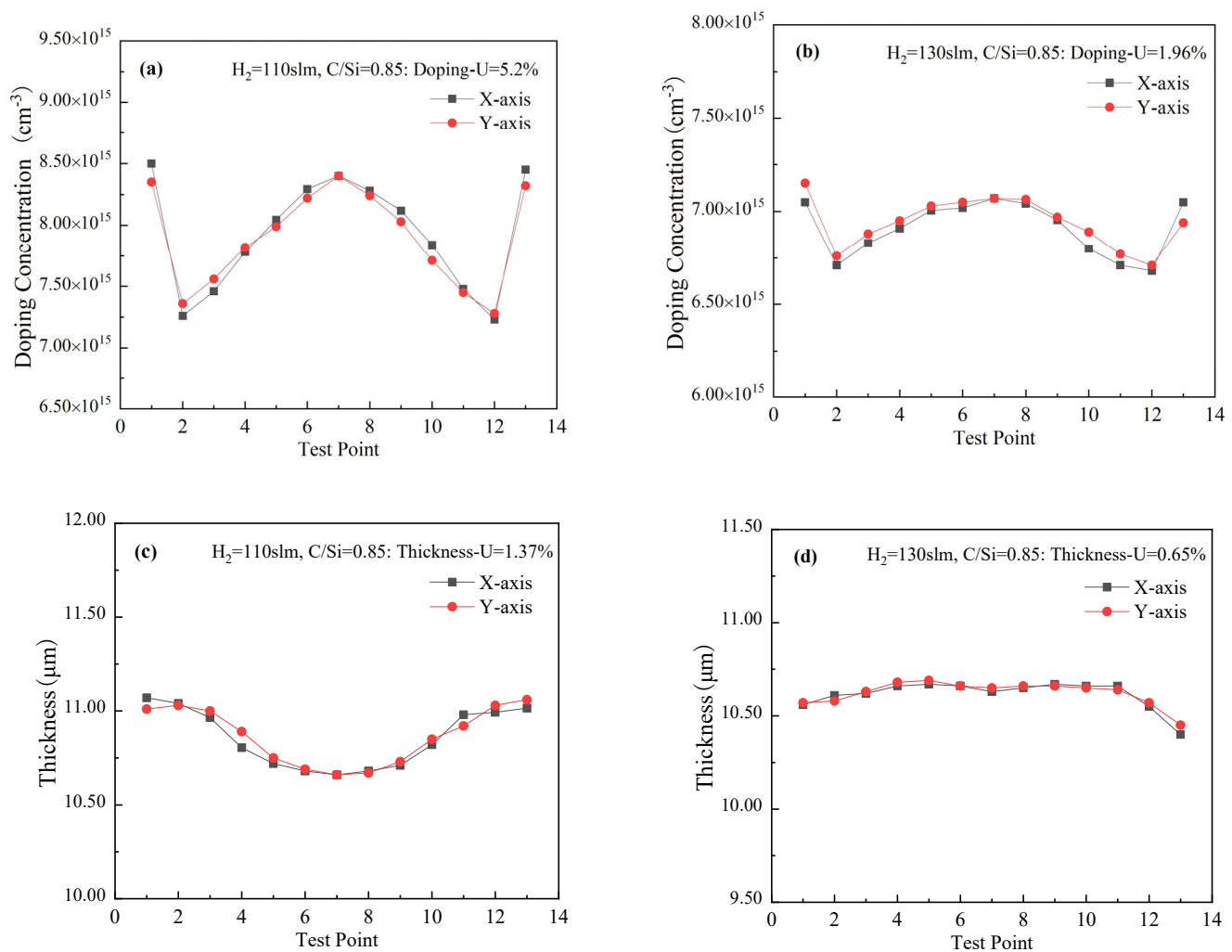
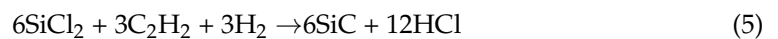


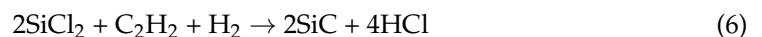
Figure 3. The doping concentration and thickness curve distribution of the epitaxial wafer at C/Si ratio of 0.85 with a carrier gas flow rate of (a,c) 110 slm and (b,d) 130 slm, respectively.

The reason for the significant improvement in doping and thickness uniformity with an increase in H_2 carrier gas flow is as follows: the gas flows horizontally from the upstream to the downstream of the chamber and passes through the substrate surface in a laminar manner. Due to different decomposition and sedimentation conditions of different sources, carbon sources (C_2H_4) have more significant “along-track depletion” rates compared to silicon sources (TCS), resulting in less of the carbon source at the center due to wafer rotation [37]. Thus, there is a decrease in the actual C/Si ratio on the wafer from the edge to the center. According to the “site-competition epitaxy” of C and N atoms [38–40], the doping concentration gradually decreases from the center to the edge on the wafer (it is worth noting that a higher doping concentration at the wafer edge is due to compensation by adding N_2 during the epitaxy process). When the chamber pressure remains constant and the carrier gas flow increases, it leads to an increased flow rate and reduced residence time of source gas inside the chamber, which slows down gas depletion along the gas flow direction, reducing differences in the C/Si ratio between the edge and center on the wafer [41,42]. In addition, in the $SiHCl_3/C_2H_4/H_2$ system, based on the numerical simulation about the 4H-SiC epitaxial chemical vapor deposition process, there are four chemical reactions, which include three gas phase reactions and one surface reaction [42–45]. These equations are shown as follows [42]:

The gas-phase reactions:



The surface reaction:



From these equations, it can be observed that $SiCl_2$ is the silicon intermediate, while C_2H_2 is the carbon intermediate with a concentration that is unaffected by the silicon precursor [44]. The chemical reaction of Equation (4) is reversible and the reverse rate cannot be ignored [44]. As the H_2 carrier gas flow increased, the consumption of C_2H_4 from upstream to downstream of the reaction chamber was slowed down, which increased the true C/Si ratio at the center of the epi-wafer. Consequently, there is a decreased difference in the surface doping concentration and thickness between the sub-edge and edge of the wafer, resulting in a significantly improved doping concentration and thickness uniformity. It is worth noting that the thickness curves can also significantly influence the ratio of gas-flow distribution among the central and lateral tunnels [41].

3.2. The Influence of the C/Si Ratio on the Surface Roughness and Doping Uniformity of the SiC Epitaxial Layer

Figure 4 illustrates the defect inspection results of two SiC epitaxial wafers subjected to identical process conditions (with a C/Si ratio of 0.95 and carrier gas flow rate of 130 slm). As depicted in Figure 4a,b, it can be seen that the defects on both epitaxial wafers are unevenly distributed. The magnified image, taken from the localized anomalous region of the epitaxial wafers (Figure 4c), reveals that this area is significantly rougher and accompanied by aggregated macroscopic steps.

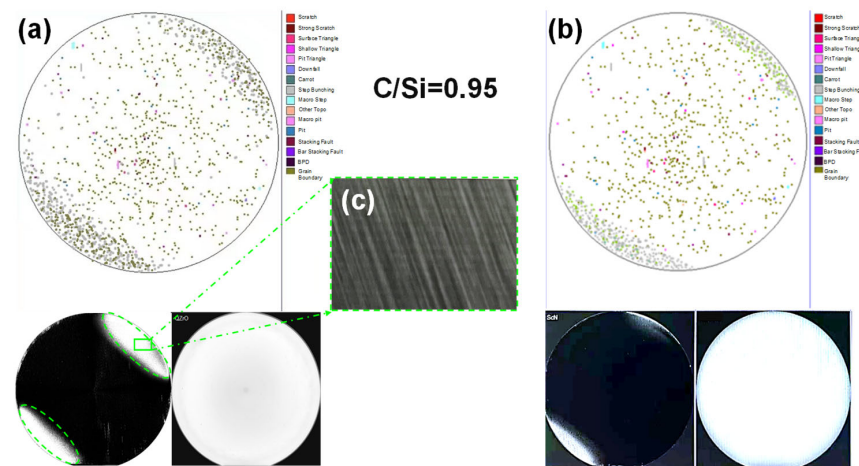


Figure 4. (a,b) are the defect inspection results of the epitaxial layer when the H_2 carrier gas flow rate is 130 slm and a C/Si ratio is 0.95; (c) is a localized magnified view at the edge of the epitaxial wafer.

The test results demonstrate the impact of reducing the C/Si ratio to 0.85 on surface defects observed on the epitaxial wafer. Notably, as depicted in the aforementioned figure, when employing the same process recipe, surface defects on both epitaxial wafers exhibit a uniform distribution without any localized aggregation as previously observed. Furthermore, upon closer examination at higher magnification (Figure 5c), it is evident that the macroscopic steps present on the epitaxial wafer's surface disappeared with a decrease in the C/Si ratio.

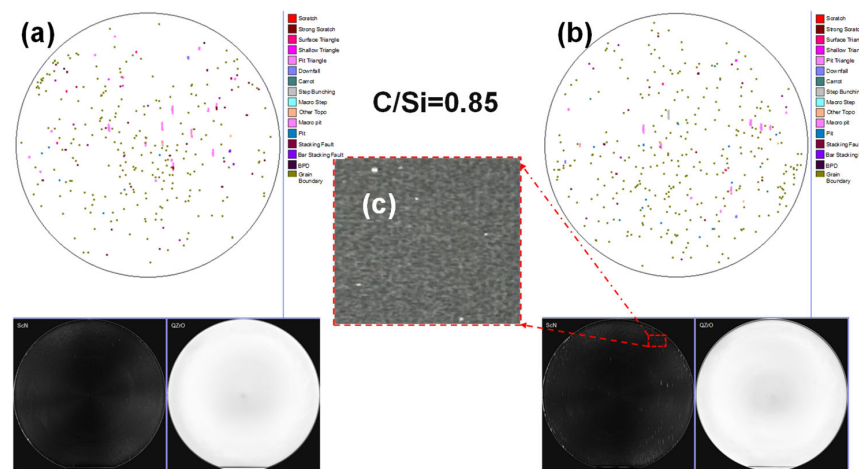


Figure 5. (a,b) are the defect inspection results of the epitaxial layer when the H_2 carrier gas flow rate is 130 slm and the C/Si ratio is 0.85; (c) is a localized magnified view at the edge of the epitaxial wafer.

The Atomic Force Microscope (AFM) images of the epitaxial wafer surface are presented in Figure 6, illustrating the impact of different C/Si ratios while keeping other process parameters constant. It is evident that the surface roughness R_a of the wafers decreases significantly from 1.82 nm to 0.16 nm as the C/Si ratio reduces from 0.95 to 0.85, with a constant H_2 carrier gas flow rate of 130 slm. A comprehensive comparison among Figures 4–6 reveals that at a C/Si ratio of 0.95, the wafer surface exhibits pronounced roughness, particularly at the edge compared to the center, due to the higher actual C/Si ratio at the edge region compared to in its central part. In SiC homogeneous epitaxy, maintaining an appropriate C/Si ratio is crucial for achieving high-quality epitaxial layers. Excessively high C/Si ratios tend to promote the “two-dimensional nucleation” growth mode during epitaxial processes, which disrupts step flow growth and ultimately leads to increased surface roughness [46]. However, when reducing the C/Si ratio to 0.85, there is a

more rapid decline in the actual C/Si ratio towards the wafer's edge relative to its center region, facilitating the step flow growth mode and resulting in significant improvement in excessive edge roughness.

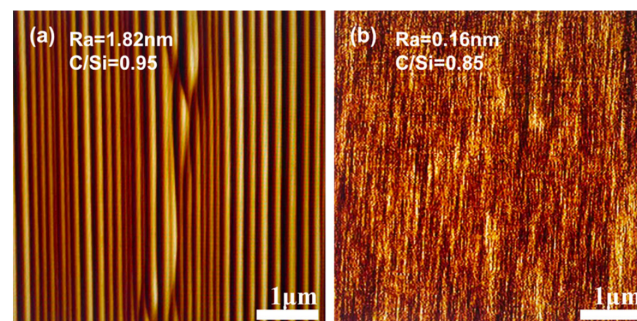


Figure 6. Surface AFM morphology at a carrier gas flow rate of 130 slm with the C/Si ratio of (a) 0.95 and (b) 0.85, respectively.

3.3. Effect of C/Si Ratio on the Doping Uniformity and the Growth Rate of the Epitaxial Layer

Figure 7 shows the doping and thickness distribution curves of the two wafers in Figure 4. From Figure 7, we can see that when the C/Si ratio is 0.95, the uniformity of doping concentration on the wafer surface is 4.2% and 4.9%, respectively. Moreover, the doping distribution curves exhibit noticeable asymmetry, indicating abnormally high doping concentrations at certain edge points. By considering the results from surface defect inspection (Figure 4), surface roughness analysis (Figure 6), and test point distribution mapping (Figure 2) presented earlier, it becomes apparent that these abnormal high-doping concentrations points occur within these areas characterized by relatively large surface roughness caused by macro-step aggregation phenomena. In other words, the aggregation of macro-steps on the wafer surface causes the doping concentration measurement anomaly, resulting in edge doping concentration asymmetry, and ultimately worsens the overall doping concentration uniformity. When the C/Si ratio decreases to 0.85 (as depicted in Figure 8), a significant enhancement in the uniformity of doping concentration is observed in the epitaxial layer, optimized from over 4% to approximately 1.8% and 1.6%, respectively. Moreover, the doping concentration distribution curves exhibit evident symmetry, displaying a characteristic “W” shape. By considering Figures 5 and 6 together, it becomes apparent that the reduction in overall roughness of the epitaxial layer on SiC substrates is primarily responsible for the improvement of symmetry.

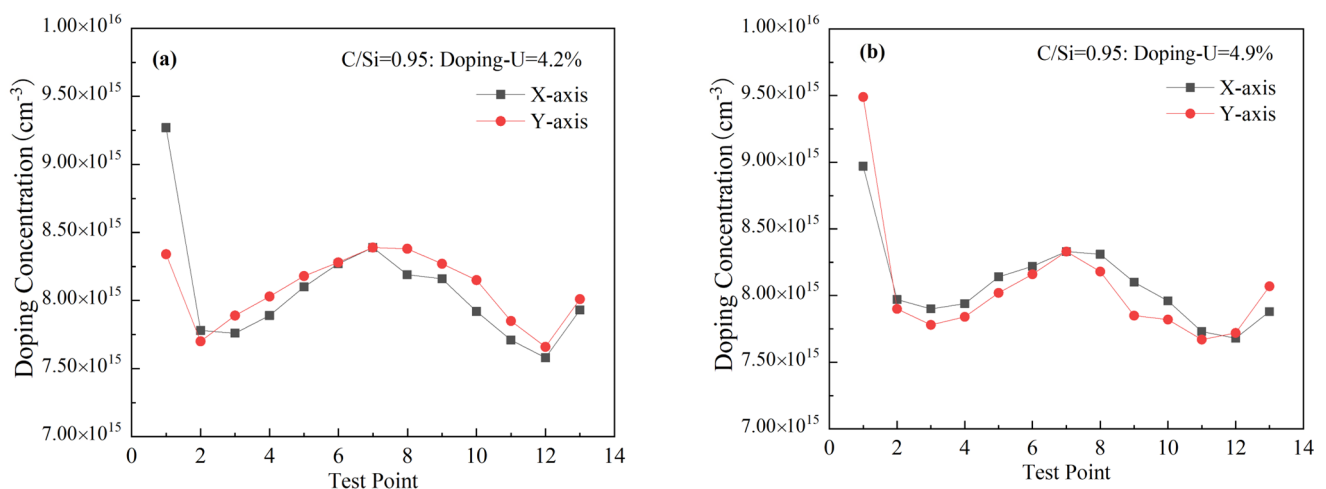


Figure 7. The doping concentration distribution curve on the surface of the epitaxial wafers with a carrier gas flow rate of 130 slm and a C/Si ratio of 0.95; the doping uniformity is (a) 4.2% and (b) 4.9%, respectively.

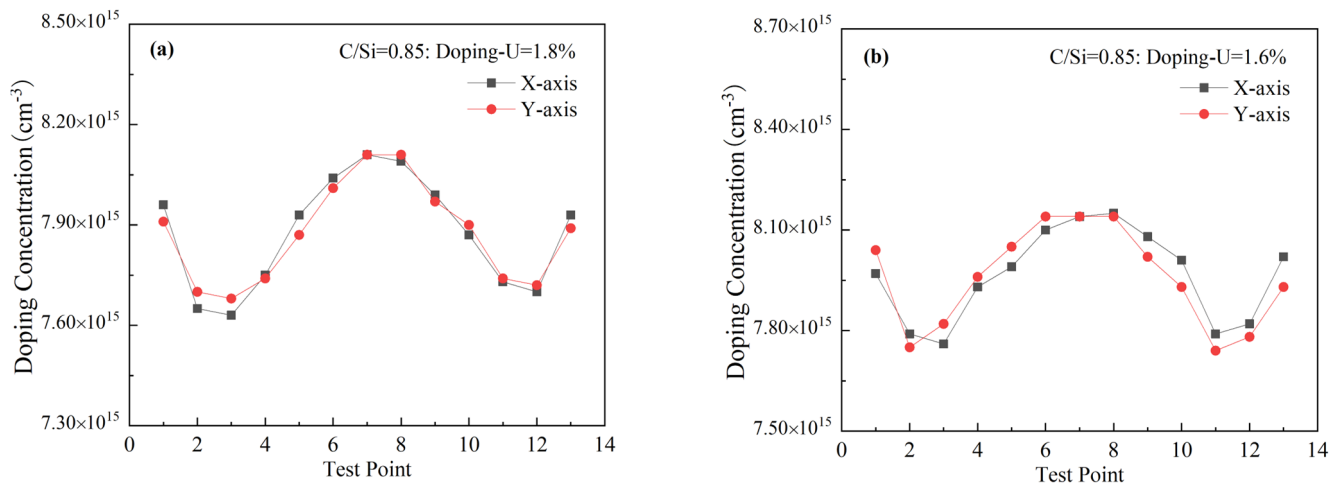


Figure 8. The doping concentration distribution curve on the surface of the epitaxial wafer with a carrier gas flow rate of 130 slm and a C/Si ratio of 0.85; the doping uniformity is (a) 1.8% and (b) 1.6%, respectively.

Figure 9 shows the thickness distribution curves of the epi-layers when the C/Si ratios were kept at 0.95 and 0.85, respectively. It can be seen that the thickness curves on the wafer are basically symmetrical. However, the overall average thickness of the epi-layers deposited on the wafer decreased from 11.19 μm to 10.87 μm , indicating that the average growth rate slowed down as the C/Si ratio reduced to 0.85, which is mainly caused by the reduction in the carbon source (C_2H_4) flow rate.

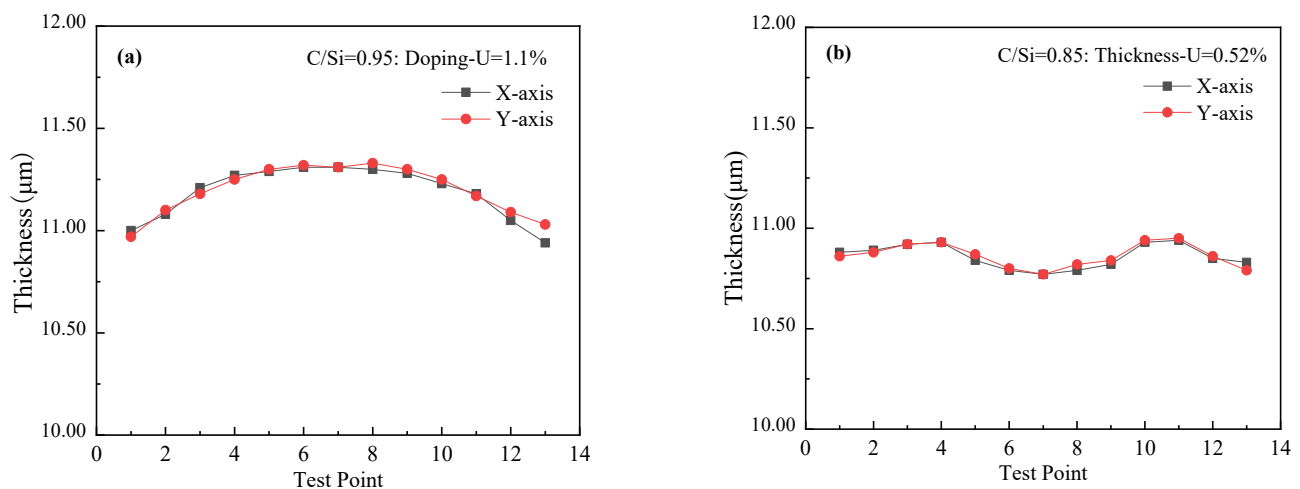


Figure 9. The thickness distribution curves on the surface of the epi-wafer at a carrier gas flow rate of 130 slm with different C/Si ratios at (a) 0.95 and (b) 0.85, respectively.

In summary, by optimizing the process parameters, i.e., increasing the carrier gas flow rate and reducing the C/Si ratio, the doping concentration uniformity of the epitaxial wafers was stabilized between 1 and 2%, and the thickness uniformity of the epitaxial wafers was stabilized at less than 1%. Additionally, the surface roughness R_a was significantly reduced to an impressive level of 0.2 nm, surpassing the industry standards established by reputable manufacturers specializing in epitaxial wafer production.

4. Conclusions

In this work, we successfully prepared high-quality SiC epitaxy wafers on domestic 6-inch 4H-SiC wafers using a self-developed SiC epitaxy growing reactor. Additionally, the effects of the carrier gas flow rate and C/Si ratio on doping concentration uniformity

and surface roughness of the epitaxial wafers were investigated. The results demonstrate that increasing the carrier gas flow rate can minimize source gas loss during deposition and enhance the doping and thickness uniformity of the 6-inch epitaxial wafer to less than 2% and 1%, respectively. Moreover, reducing the C/Si ratio significantly suppresses the “two-dimensional nucleation growth mode” and optimizes the surface roughness Ra of the epitaxial wafer to below 0.2 nm, meeting industry standards for high-quality SBD and MOSFET devices.

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