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Influence of Temperature and Flow Ratio on the Morphology and Uniformity of 4H-SiC Epitaxial Layers Growth on 150 mm 4° Off-Axis Substrates

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Abstract: The homoepitaxial growth of 4H-SiC films was conducted on 4H-SiC 150 mm 4° off-axis substrates by using a home-made hot-wall chemical vapor deposition (CVD) reactor. Special attention was paid to the influence of the growth temperature on the surface morphology, growth rate, doping efficiency, and structural uniformity of the films. Among the above factors, growth temperature and flow ratio were shown to be the essential parameters to produce high-quality homoepitaxial layers. Furthermore, a two-side flow tunnel was introduced to control the growth temperature nonuniformity in the reactor. The influence of flow ratio on the epitaxial layer uniformity was also studied. It was found that the surface roughness increased with the increasing temperature, achieving its minimum value of 0.183 nm at 1610 °C. Besides that, the film growth rate decreased with the increase in growth temperature, whereas the degrees of thickness non-uniformity, N_2 doping non-uniformity, and doping efficiency increased. Meanwhile, both the thickness and doping uniformity can be improved by adjusting H_2 and N_2 flow ratios, respectively. In particular, the use of the H_2 ratio of 1.63 and N_2 ratio of 0.92 enabled one to increase the degree of uniformity of thickness and doping by 0.79% (standard deviation/mean value) and 3.56% (standard deviation/mean value), respectively, at the growth temperature of 1630 °C.

Keywords: 4H-SiC homoepitaxial layer; 150 mm 4° off-axis substrate; CVD; growth temperature; flow ratio; uniformity

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1. Introduction

Over the past few years, silicon (Si)-based power devices have reached their physical limits in terms of the capability to block high voltage, provide low on-state voltage drop, and switch at a high frequency. As one of the representative semiconductor materials with a wide band-gap, 4H-silicon carbide (4H-SiC) has many promising features such as high thermal conductivity, high electric breakdown field, and high saturation velocity, which cause it to be a suitable next generation material for high-voltage power devices [1–3]. Chemical vapor deposition (CVD), which used to grow 4H-SiC epitaxial layers on homogeneous substrates, is a powerful technique to produce high-quality drift layers of 4H-SiC power devices [4,5]. Meanwhile, the epitaxial film growth by CVD depends on the high-temperature reaction between the reactants and the flow status in the reactor [6,7]. In such conditions, the temperature and distribution of the gas flow ratio that carries reactants are the key factors, especially for the large diameter wafers, affecting the surface morphology,

thickness uniformity, doping concentration, and defects of the epitaxial film. So far, the homoepitaxial growth of 4H-SiC layers has been performed on the off-axis substrates to facilitate the "step-flow mode" [8] and ensure good crystallinity. For instance, the epitaxial layer growth on 4H-SiC substrates with an off-cut angle of 8° (from [0001] to [11$\bar{2}$0]) yields fewer surface defects, but a large off-angle would raise cost of the material. Since less 4H-SiC substrates can be sliced from the crystal boule by using off-cut 8° substrate, the 4° off-axis 4H-SiC substrate was used as the cost-saving solution [9]. In addition, the density of basal plane dislocations (BPDS) in the epilayers was reduced by applying the 4° off-axis 4H-SiC substrate [10,11].

The epitaxial growth of 4H-SiC has aroused great interest from researchers because of surging development of SiC power devices in recent years. As mentioned above, the surface morphology, thickness uniformity, and doping concentration are three necessary factors to judge the quality of epitaxial layers. The earlier study focusing on 4H-SiC homoepitaxially grown on a 2-inch on-axis substrate [12] revealed that a good quality film surface morphology was achieved on a (000$\bar{1}$) C-face on-axis substrate. The epi-layer growth on 4H-SiC 4° off-axis (0001) Si-face and (000$\bar{1}$) C-face substrates has been also reported [13]. It was shown that the Si-face epi-layer, with the immense step-bunching, was obtained under C-rich conditions, whereas the C-face epi-layer with high quality morphology was grown in a broader range of C/Si ratio at 1600 °C. Moreover, the thickness uniformity can be improved for epitaxially grown films on 35 mm wafers by increasing the hydrogen carrier gas and height of the gas flow tunnel [14]. The use of chlorine-based gas and in-situ etching were conducive to the rapid growth of a 4H-SiC epi-layer with a smooth surface and good uniformity on an off-angle substrate [15–17]. Various experiments on 2-inch and 4-inch substrates demonstrated that the silicon droplet on the epitaxial layer can be suppressed by increasing the growth temperature [18]. It is found that the C/Si ratio is an essential parameter to obtain high-quality 4H-SiC epitaxial layers, whose growth rate increases with the increase in the C/Si ratio [19]. Additionally, the impact of doping on the SiC epilayers onto a 3-inch substrate has been studied showing that the hydrogen flow is the main factor affecting the doping uniformity [20].

With the continuous development of SiC power devices, the large-size substrates (150 mm) have become a primary way for many manufacturers to reduce power device costs [21,22]. The large-size substrate has become a primary way for manufacturers to reduce the costs to produce power devices. Meanwhile, as the diameter of the substrate increases, the temperature gradient within the substrate during the CVD process also increases. Besides, the distribution and depletion of the reactant flow changes drastically compared to that on the smaller substrate and obtaining a high-quality epitaxial layer on a large-size substrate is a big challenge. To ensure device performance stability in the industry, the surface roughness, surface defects, and epitaxial layer uniformity on large-size (150 mm) substrates has been investigated by many groups [23–29]. However, these studies mainly focused on the impact of the C/Si ratio, rotation speed, and in-situ etching on the quality of the epitaxial layer in the chlorine-based growth system. In turn, the influence of growth temperature on the surface roughness was scarcely investigated. Regardless of some reports analyzing the macroscopic images on 4-inch substrates [30], the impact of 150 mm substrates on the microstructural surface roughness, growth rate, and nitrogen-doping efficiency of epitaxial layers is still questioned. In addition, few attempts have been made to adjust the flow ratio in order to achieve good uniformity in both the thickness and nitrogen doping of epi-layers.

In this paper, 4H-SiC epitaxial layers were grown onto 150 mm 4° off-axis substrates via a low-pressure CVD method. Particular attention was paid to the effect of the growth temperature on the surface roughness, epitaxial layer thickness (growth rate), and doping efficiency. In addition, the influences of the main hydrogen flow ratio and nitrogen flow ratio on the thickness uniformity and doping uniformity were investigated by conducting single-variable experiments and the optimal uniformity was obtained by simultaneously varying both flow ratios.

2. Experiments and Methods

The homoepitaxial growth of 4H-SiC layers was carried out in a low pressure home-made horizontal hot-wall CVD reactor using a $SiHCl_3$ (TCS) + C_2H_4 + H_2 system. The 150 mm n-type 4H-SiC substrates used in this work were purchased from Semisic Crystal Co., Ltd., Shanxi, China. The substrates had Si-terminated (0001) faces with the 4° off-axes oriented toward [11$\bar{2}$0] the crystal direction. TCS and ethylene (C_2H_4) served as the Si source and C source and H_2 was used as the dilution, carrier, and etching gas. Before the reactor reached the growth temperature, the H_2 gas was introduced for in-situ etching of the SiC substrate for 10 min. There are a total of 9 epitaxial runs during the whole experiment. Within the growing process, the H_2 flow rate and C/Si ratio were fixed at 120 slm and 1.1, respectively, under the chamber pressure of 100 mbar. The growth time was 30 min and the growth temperature was varied from 1610 °C to 1680 °C. The rotational speed of the holder with the SiC substrate was around 50 rpm. Besides, the nitrogen doping was performed at a fixed flow rate of 250 sccm. As shown in Figure 1a,b, due to the large size (150 mm) of the substrate, two-side flow tunnels were introduced in the reactor chamber. The H_2 and N_2 flow ratios were adjusted from 1.5 to 2.3 and from 0.92 to 1.7, respectively, by changing the proportions of the mass flow controller (MFC) between the main flow rate and side flow rate (Main/Sides).

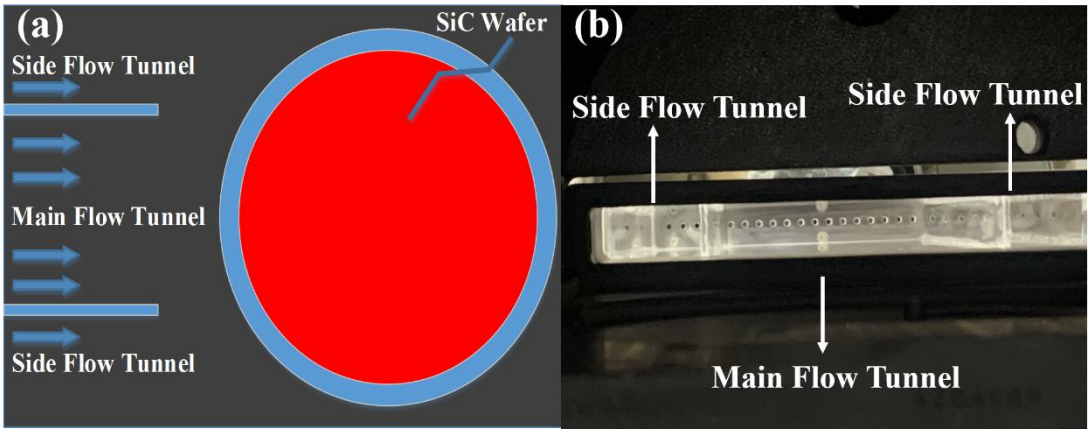


Figure 1. Flow tunnel image of CVD reactor chamber: (**a**) Schematic image, (**b**) Real image.

Figure 2a displays an epitaxially grown 150 mm 4H-SiC wafer obtained using epitaxial growth during the experiments.

Seventeen points were chosen on the wafer surface for the detail of Figure 2b. Among them, point 5 is at the center of the wafer and the distance between the test points is 17.5 mm. Points 1, 9, 10, and 17 are 5 mm from the edge of the wafer. The points were chosen to lie along the two lines perpendicular to each other. The thickness and doping concentration were evaluated using Fourier transform infrared spectroscopy (FTIR) and mercury capacitance–voltage (MCV) systems, respectively. The overall thickness, doping concentration, and uniformity of the whole epitaxial wafer were calculated by using the mean value ($\bar{x}$) and standard deviation/mean value ($\sigma/\bar{x}$) of these seventeen points data as can be seen in Equations (1) and (2). The surface roughness and surface morphology were studied at center of the wafer (test point 5) using an atomic force microscope (AFM) within the area of 10 μm × 10 μm.

$$\text{Mean Value} = \bar{x} = \frac{\sum_{i=1}^{17} x_i}{17} \quad (1)$$

$$\text{Uniformity} = \sigma/\bar{x}\ (\%) = \frac{\sqrt{\frac{\sum_{i=1}^{17}(x_i-\bar{x})}{16}}}{\bar{x}} \quad (2)$$

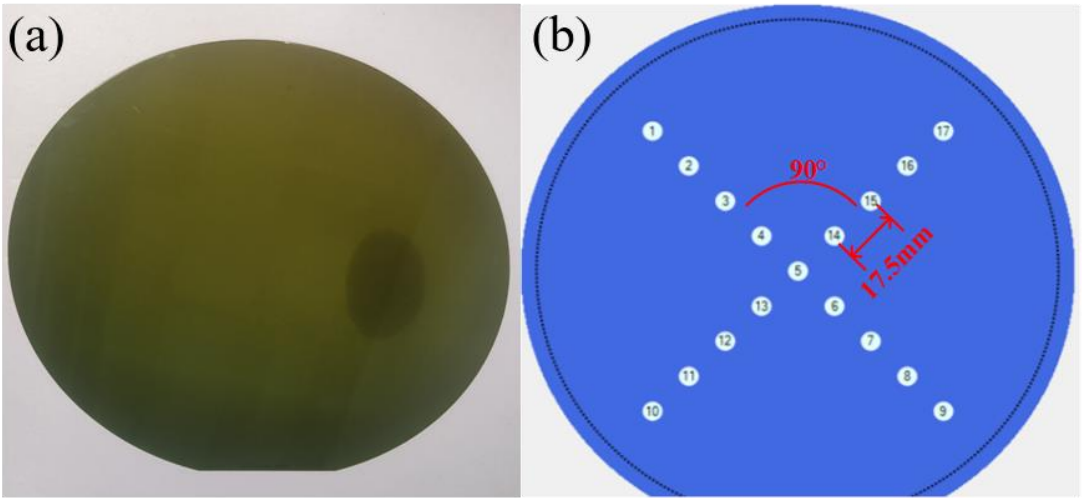


Figure 2. 150 mm 4H-SiC wafer: (**a**) wafer after epitaxial growth, (**b**) seventeen test points on the wafer surface.

3. Results and Discussion

3.1. Growth Temperature

The growth temperature is an important parameter during the epitaxial growth process. In order to study the effect of temperature on the quality of the epitaxial layer and avoid the impact of the flow ratio, the experiments were carried out at the H_2 flow ratio (Main/Sides) of 1.5 and the N_2 flow ratio (Main/Sides) of 1.28. In the meantime, the temperature was changed from 1610 °C to 1680 °C. Figure 3a–d display the two- (2D) and three-dimensional (3D) AFM images of the epitaxial wafer produced under the above conditions. At the lower temperature, the surface of the wafer was smooth and flattened. On the other hand, with the increasing temperature, the giant waves appeared, and the surface gradually became rougher. As a result, the surface roughness drastically increased from 0.183 nm (1610 °C) to 0.721 nm (1680 °C). Similar results were reported on the 4-inch 4H-SiC homoepitaxial layer [30]. The appearance of a giant wave and severe surface roughness at a higher growth temperature was attributed to a step bunching phenomenon that became more and more obvious [31]. In addition, the gradually increased temperature enhanced the migration of atoms on the surface of the SiC substrate [32] and would also cause a rougher surface at higher growth temperatures.

It can be seen from Figure 4a that the thickness or growth rate has decreased with the increase in temperature. At the highest temperature (1680 °C), the thickness of the epitaxial layer decreased to 14.156 µm. Similar results concerning the temperature effect on the growth rate have been reported in previous studies [33,34]. With the rising of the temperature, the impact of H_2 etching would be remarkable, affecting the epitaxial layer's growth rate to some degree [34]. On the contrary, the thickness uniformity exhibited an opposite tendency: as the temperature increased to 1680 °C, the thickness uniformity reached 6.5% (standard deviation/mean value). With the increase in the growth temperature in the reactor chamber, the temperature distribution became more and more uneven, causing the great difference in the growth rates at various areas of the wafer surface and consequently affecting the thickness uniformity of epitaxial layer during the growth process to a large extent.

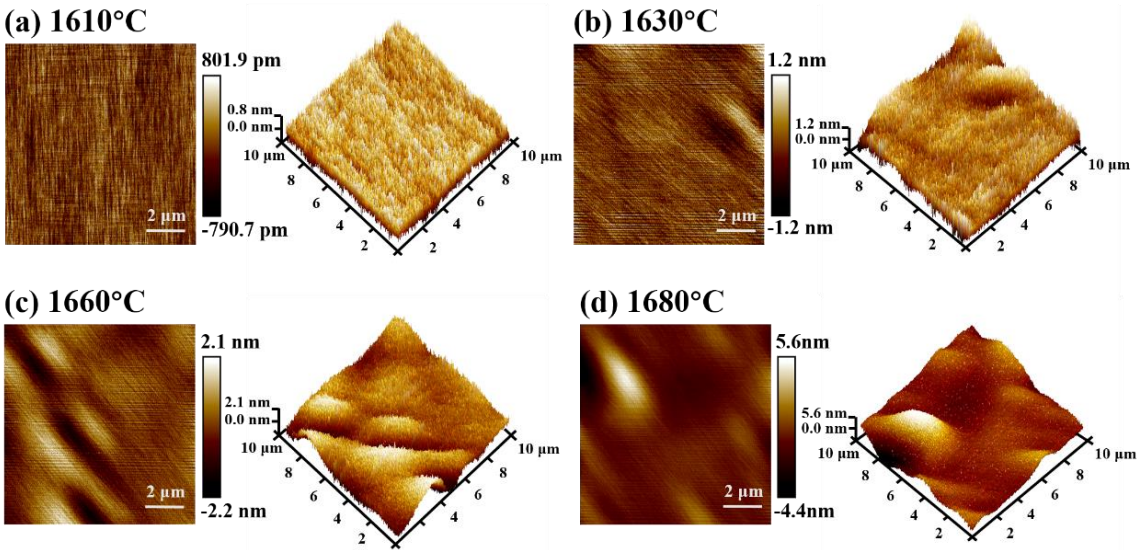


Figure 3. 2D and 3D AFM images of 4H-SiC wafers at the growth temperature of: (**a**) 1610 °C, (**b**) 1630 °C, (**c**) 1660 °C, and (**d**) 1680 °C.

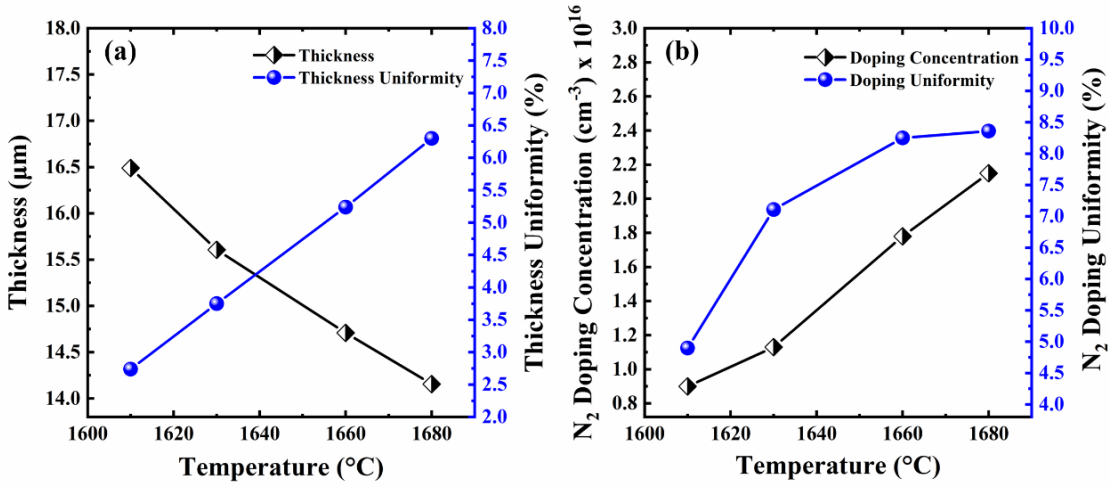


Figure 4. Thickness and doping concentration at various temperatures: (**a**) thickness and thickness uniformity, (**b**) N_2 doping concentration and doing uniformity.

As shown in Figure 4b, the higher the temperature, the higher the N_2 doping efficiency in the epitaxial wafer is. A similar relationship has been reported, revealing that the nitrogen incorporation could be limited by the thermally activated process [35]. The N_2 doping was quite uniform at a temperature of 1610 °C (4.9%, standard deviation/mean value), but exhibited nonuniformity at the higher temperature of 1680 °C (8.36%, standard deviation/mean value).

As mentioned above, with the increase in the growth temperature, the temperature distribution would have become more and more uneven, causing a huge difference on the doping efficiency at different areas of the wafer surface. The larger temperature difference at the higher growth temperature in the reactor chamber suggested the higher doping nonuniformity on the wafer, which explained the tendency observed in Figure 4b to some degree.

3.2. Gas Flow Ratio

To investigate the influence of the H_2 flow ratio on thickness uniformity, the experiment was further conducted under the growth temperature of 1630 °C and the N_2 flow ratio fixed at 1.28. The main and side flow tunnel were introduced into the reactor chamber to compensate for the effect of the wafer's temperature difference between the central and lateral areas. The H_2 flow ratio was varied from 1.5 to 2.3 to observe the thickness change from point 1 to point 9. As shown in Figure 5a, the thickness curve had a sunken shape under the flow ratio of 1.5 but changed to the W-like shape at the flow ratio of 1.63. As the flow ratio changed to 1.94 and 2.3, the curve became bulged. It was thus suggested that the thickness of the central area increased with the increase in the main flow rate of H_2.

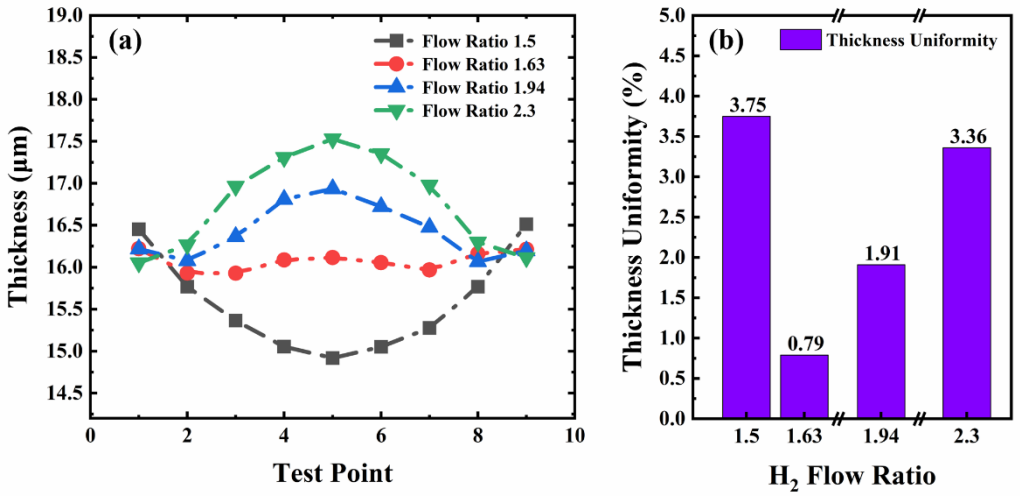


Figure 5. Influence of H_2 flow ratio on: (**a**) radial distribution and (**b**) uniformity of the wafer thickness.

The thickness uniformity can be effectively improved by adjusting the flow ratio of H_2. Figure 5b depicts the variation in thickness uniformity of the epitaxial layer and the optimal thickness uniformity of 0.79% (standard deviation/mean value) is obtained at the H_2 flow ratio of 1.63. This result indicates that the proportions of Si and C sources for the central and side areas have changed due to the equivalently adjusted flow ratio of the carrier gas H_2.

The influence of the N_2 flow ratio on the doping concentration was also investigated at the temperature of 1630 °C and the H_2 flow ratio was fixed at 1.63. According to Figure 6a, the N_2 concentration distribution exhibited the "W" shape at the N_2 flow ratio of 0.92. In turn, the N_2 doping concentration curves at the N_2 flow ratio of 1.7 and 1.28 had a bulge shape and the side areas in the first case were smaller than those at the N_2 flow ratio of 1.7. Figure 6b displays the influence of the N_2 flow ratio on the doping uniformity. As seen from the plot, the optimal uniformity of 3.56% (standard deviation/mean value) was achieved at the N_2 flow ratio of 0.92. These results imply that the distribution of the N_2 doping concentration within the corresponding areas can be changed accordingly by adjust the N_2 flow ratio.

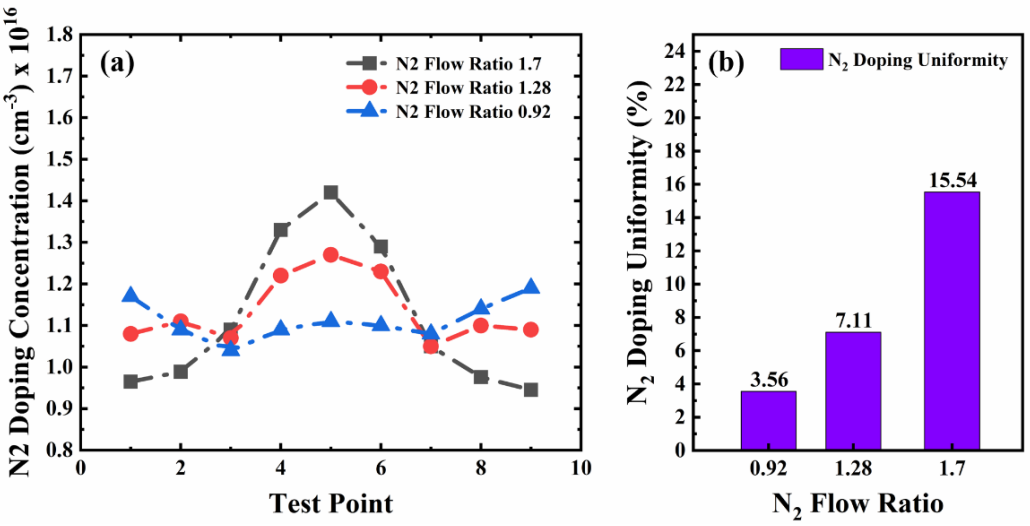


Figure 6. Influence of N_2 flow ratio on (**a**) radial distribution of doping concentration and (**b**) doping uniformity of the wafer.

4. Conclusions

The 4H-SiC homoepitaxial layers were grown on the 150 mm 4° off-axis substrates using a home-made horizontal hot-wall CVD reactor. The effects of the growth temperature, H_2 flow ratio, and N_2 flow ratio on the surface roughness and uniformity of large-size 4H-SiC epitaxial layers were investigated by changing the growth temperature and flow ratio. The AFM, FTIR, and MCV studies revealed that the change in the growth temperature affected the surface roughness, morphology, growth rate, and uniformity of the epi-layers. Moreover, the nonuniformity of the epitaxial layer due to the temperature differences on the wafer surface could be compensated by adjusting the H_2 and N_2 flow ratio. With the increase in temperature, the surface roughness increased, so that the minimum surface roughness value was achieved at 1610 °C. Meanwhile, the growth rate, thickness uniformity, and doping uniformity decreased, whereas the efficiency of N_2 was enhanced with the increasing temperature. In particular, the optimal thickness uniformity of 0.79% (standard deviation/mean value) was obtained at a flow ratio of 1.63 and the best N_2 doping uniformity of 3.56% (standard deviation/mean value) was acquired at the N_2 flow ratio 0.92. In both cases, the temperature was fixed at 1630 °C. Thus, the growth temperature and flow ratio are two important parameters in the homoepitaxial growth of high-quality 4H-SiC layers on 150 mm 4° off-axis substrates.

Author Contributions: Z.T.: Methodology, Data curation, Writing—original draft, Formal analysis. L.G.: Validation, Investigation, Formal analysis, Data curation. H.M.: Conceptualization, Supervision, Funding acquisition, Project administration, Writing—review and editing. C.M.: Validation, Investigation. S.W.: Supervision, Resources. N.Z.: Investigation, Writing—review and editing. J.H.: Data curation, Writing—review and editing. J.F.: Project administration, Investigation, Supervision. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The data that support the findings of this study are available from the corresponding author, Z.T. and H.M., upon reasonable request.

Conflicts of Interest: The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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