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Effect of C/Si Moore Ratio on the Morphology and Crystallinity of 3C-SiC/Si(100) by the Modified Two-Step Carbonization Process

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A modified two-step carbonization process was used to grow high oriented 3C-SiC films on Si (100) substrates by using SiH_4 and C_3H_8 as precursors. The investigation revealed that the C/Si Moore ratio has important influence on the SiC nucleation model on Si substrates and the crystal quality of 3C-SiC films. The effect of C/Si Moore ratio on the SiC/Si (100) were evaluated by Microscopy and SEM. This indicated that the growth model of the individual nuclei gradually changed from vertical to lateral growth as the C/Si Moore ratio increased. High-resolution X-ray diffraction and Raman spectra were used to analyze the crystalline quality of 3C-SiC films grown at 1385 °C under different C/Si ratios. The results showed that the optimum C/Si Moore ratio for high oriented and high crystal quality 3C-SiC films was 1.6. The mirro-like high-crystallinity 3C-SiC films on the Si (100) are grown with a 1.6 C/Si ratio at 1385 °C for 1hr. XRD results showed that the diffraction intensity of 3C-SiC (200) peak was very closed to that of Si (400) peak and the FWHM value of it was about 0.19°. The surface roughness was 3.6 nm.

Keywords: 3C-SiC films, C/Si ratio, morphology, modified two-step carbonization process.

1. INTRODUCTION

Silicon carbide (SiC) crystal materials have been attended to the superior wide band material. Cubic silicon carbide (3C-SiC), as the common polytype of numerous SiC polytypes, can been grown on Si single crystal wafer even there is a big lattice mismatch. It can be applied in a wide range of fields, because of its lower cost, lower lattice mismatch with nitrides and graphene [1, 2]. In addition, as an isotropic crystalline, 3C-SiC has inherent advantages in device operation compared with highly anisotropic hexagonal polytypes [3]. 3C-SiC can been used as a lattice transition layer between GaN and Si, which can prevent the melt-back etching effect. However, the significant mismatch in the lattice coefficient (~ 20 %) and thermal expansion coefficient (CTE, ~ 8 %) that exists between 3C-SiC and Si are two of the main reasons why it is difficult to grow a high-quality hetero-epitaxial 3C-SiC layer on Si substrate [4]. High quality 3C-SiC films could not be grown on Si substrates, until Nishino et al introduced the carbonization process [5]. Supported by the researches of Wang, L. and Cordier, Y. [6, 7], the thickness, surface morphology, off-cut angle, curvature magnitude, and shape of 3C-SiC/Si template will affect the strain relaxation and dislocation density reduction in the GaN layer which grown on the 3C-SiC/Si substrate. The growth temperature has direct effect on the surface, the crystalline quality and the curvature of 3C-SiC films. Wilhelm, M. et al. [8], Chung G.S. et al. [9], and Anzalone R. et al. [10] have grown 3C–SiC films on Si substrates at 1200 °C, 1350 °C and 1370 °C respectively. The crystalline quality and surface morphology of 3C-SiC films were the most important factors that affected manufacture of 3C-SiC/Si devices [11]. However, even though significant progress has been carried out in the past decade in 3C-SiC films procedures, the growth processes of high quality 3C-SiC films are still being studied to improve crystalline quality and surface morphology. The crystallinity, thickness, and morphology of 3C-SiC films have been found to be a strong function of the hydrocarbon concentration in the gas stream and the growth pressure [12]. Compared to sputter deposited method, the chemical vapor deposition (CVD) method is an important process, which is appropriate for layers with a variable chemical composition ratio [13, 14]. The CVD method is a commonly useful method for the growth of 3C-SiC films on Si. Until now, surface quality, crystallinity, and defects (such as the interface voids, dislocation, and grains) were still the hot research topic [15, 16]. According to the report of prof. Su, J.F. [17], the high crystallinity and void-free 3C-SiC films could be obtained by the modified two-step carbonization method.

In this work, 3C-SiC films grow on Si (100) substrates via a modified two-step carbonization method and the effects of C/Si Moore ratio on the structure and morphology of 3C-SiC films were studied.

2. EXPERIMENTAL DETAILS

Low pressure chemical vapor deposition method was used to grow the 3C-SiC films on the (100) oriented p-type Si substrates. The growth experiments were carried out using a VP508 hot-wall CVD reactor manufactured by Aixtron SE. High-purity hydrogen that was purified via a metal palladium composite membrane served as the carrier gas during the CVD procedures. The carbon and silicon precursors were provided by C_3H_8 (100%) and SiH_4 (100%), respectively. This study also used planar p-type

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Si (001) samples that were diced into 20 mm × 20 mm die. Before the start of growth, the substrates were cleaned through a standard cleaning procedure developed by the Radio Corporation of America.

The growth procedure consists of the following steps: in-situ cleaning, carbonization (step 1), carbonization (step 2), and 3C-SiC growth, as illustrated in Fig. 1. First, the substrate was heated up to 1100 °C in the hydrogen atmosphere and held at that temperature for 5 minutes to remove the silica layer and the surface contamination. During the entire modified two-step carbonization process, the H_2 flow rate was kept a constant of 30000 sccm. Then, the substrate was cooled down to around 600 °C. After that, the propane was introduced in to the reactor with a C_3H_8 molar fraction of 3.0×10^{-4}. The chamber temperature was increased to 1185 °C as quickly as possible and held for 1 minute to carry out carbonization. Then, a small SiH_4 flow was introduced to the reactor. The experiment was performed by ramping to growth temperature using a gas mixture composed of H_2, SiH_4, and C_3H_8. The C_3H_8 and SiH_4 molar fractions were 3.0×10^{-4} and 2.0×10^{-4}, respectively. After the temperature arrived at the setting growth temperature, the propane and silane sources were shut off to perform H_2 etching for 1 minute. Then, the 3C-SiC films were grown at the setting temperature for 1 h. The growing pressure was 100 mbar. In this paper, to obtain a range of C/Si Moore ratio between 0.8 and 3.0, the C_3H_8 flow rate was kept a constant of 6 ml/min but the SiH_4 flow rate was changed from 6 ml/min to 22.5 ml/min. Finally, the samples were cooled to room temperature in an argon atmosphere.

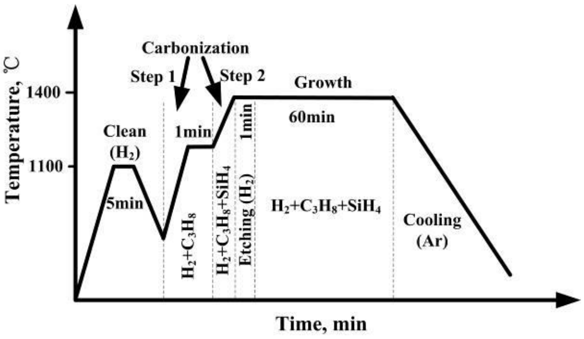
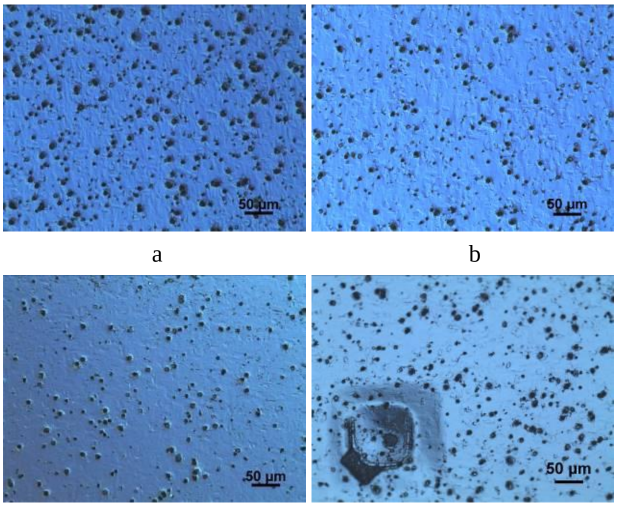


Fig. 1. Diagrammatic picture of 3C-SiC grown on the Si (100) substrates by the modified two-step carbonization method

High resolution X-ray diffractomety (Bruker D8 Discover system) was used to characterize the crystallinity of 3C-SiC films on Si. Microscopy (Olympus BX51), an atomic force microscope (Bruker Dimension Edge, Maximum scanning range XYZ: 90 um × 90 um × 10um), and scaning electron microscopy (ZEISS MERLIN Compact, magnification range = 12 – 2.000.000×) were utilized to evaluate the surface quality and thickness of the 3C-SiC films. The structural characterization was performed using micro-Raman spectroscopy (Horiba LabRAM HR800, $\lambda_0 = 514.4$ nm, laser power = 0.1 mW on sample, laser diameter size < 1 μm, resolution < 0.65 cm^{-1}).

3. RESULTS AND DISCUSSION

First, the process temperature was varied within the temperature range of 1350 – 1400 °C. 100 mbar was chosen as the most favorable growth pressure. The C_3H_8 and SiH_4 molar fractions were 2.0×10^{-4} and 4.33×10^{-4}, respectively. The C/Si Moore ratio was about 1.4. Fig. 2 shows four optical photographs of 3C-SiC films, which were grown at 1350 °C, 1370 °C, 1385 °C, 1400 °C, respectively. In Fig. 2 a and b, Many spots appeared on the surface of 3C-SiC films. Non-specular layers were obtained at temperatures lower than or equal to 1350 °C, suggesting that the saturation point had been reached. The number of the spots decreased as the growth temperature increased. However, 1400 °C is near the melt point of silicon substrate. In Fig. 2 d, when growing the 3C-SiC film, part area of silicon substrate begins to melt. The surface morphology of Fig. 2 b and c was very similar. However, through the Photoshop analysis by setting the threshold levels parameter to 80, the spots area fraction was only 4.41 % in Fig. 2 c, which was smaller than the 5.43 % in Fig. 2 b. So 1385 °C was chosen to be constant for the continued experiments.



a b

c d

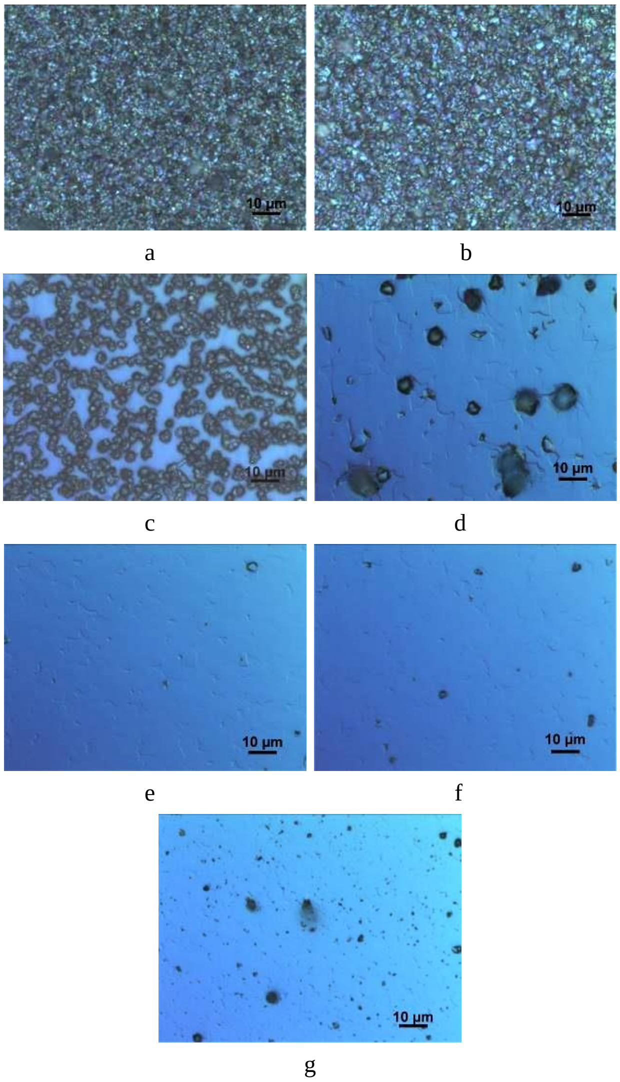
Fig. 2. Optical photographs of 3C-SiC films grown under varied temperatures for 1 h with 1.4 C/Si Moore ratio: a – 1350 °C; b – 1370 °C; c – 1385 C; d – 1400 °C (magnification = 200×)

To study the effect of C/Si Moore ratio on the quality of 3C-SiC layers, different C/Si Moore ratios were designed to grow the 3C-SiC layers. The growth temperature of 1385 °C was chosen as the most favourable parameter. The 3C-SiC layers were growth at this temperature for 1 h. The C_3H_8 molar fraction was kept constant at 2.0×10^{-4}. The SiH_4 molar fractions changed from 2.0×10^{-4} to 7.5×10^{-4}. Table 1 shows the detailed parameters.

Table 1. Typical experimental parameters for each step during growth

C/Si Moore ratio	0.8	1.0	1.2	1.4	1.6	2.0	3.0
C_3H_8 flow, ml/min	6	6	6	6	6	6	6
SiH_4 flow, ml/min	22.5	18	15	13	11	9	6
H_2 flow, sccm	30000						

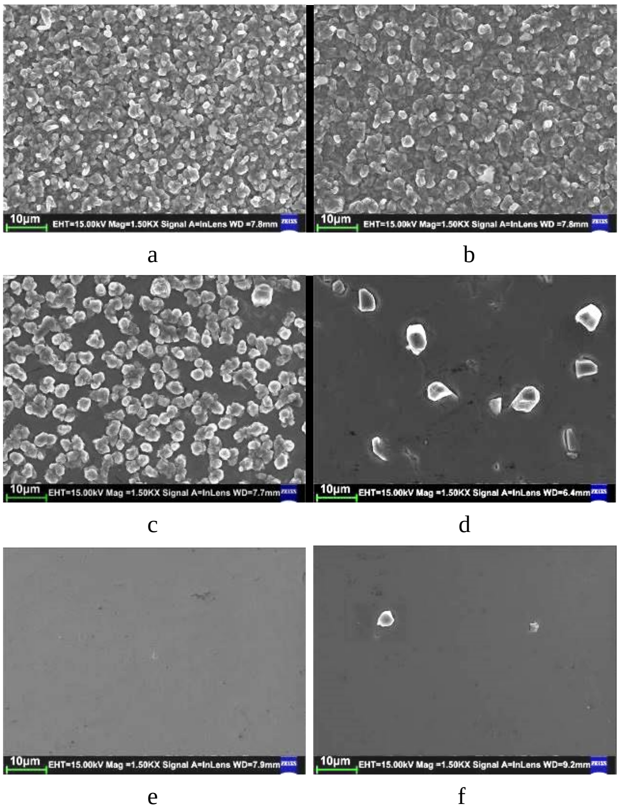
Fig. 3 shows photographs of 3C-SiC films, which were grown at 1385 °C for 1 h with varying C/Si Moore ratios. It is apparent that the C/Si Moore ratio dramatically affected film morphology. When the C/Si Moor ratio was less than 1.6, many spots appeared on the surface of the 3C-SiC layers. According to the results, the number of the spots increased as the C/Si Moore ratio decreased. A careful observation by SEM shows they are surface protrusions, which can be observed from Fig. 4. As the C/Si Moore ratio increased to more than 1.6 (including 1.6), the number of the protrusion type defects was dramatically decreased. A specular 3C-SiC film surface was obtained.



a b c d e f g

Fig. 3. Optical pictures of 3C-SiC films which grown at 1385 °C for 1hr with different C/Si Moore ratios: a – C/Si = 0.8; b – C/Si = 1.0; c – C/Si = 1.2; d – C/Si = 1.4; e – C/Si = 1.6; f – C/Si = 2.0; g – C/Si = 3.0 (magnification = 1000 ×)

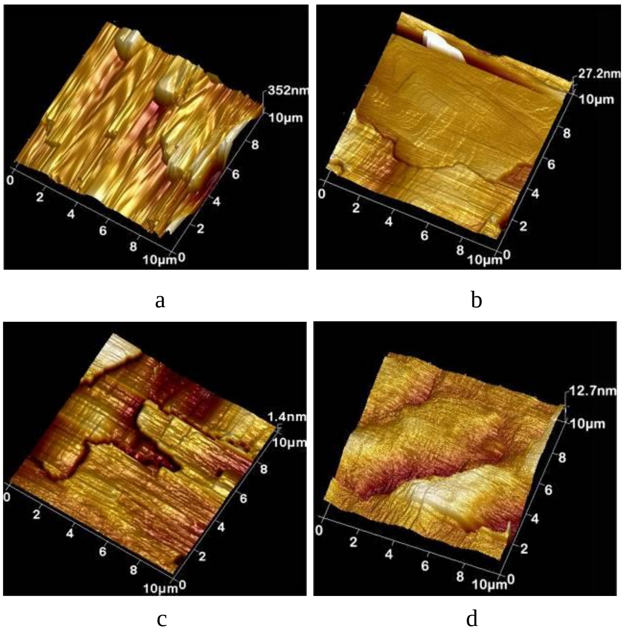
In this paper, SEM analysis was used to study the surface quality and the microstructure of 3C-SiC films on Si (100) substrates. Fig. 4 shows the typical SEM images of as-grown 3C-SiC films on Si (100) grown at 1385 °C with different C/Si Moore ratios. When the C/Si Moore ratio was less than 1.0, small 3C-SiC grains covered the entire surface of the sample. As the C/Si Moore ratio increased, the quantities of the 3C-SiC grains on the surface of the films gradually decreased. The surface morphology of 3C-SiC films changed to smooth mosaic-like surface from rough grain-like surface, significantly. For C/Si ≥ 1.6, the surface topography was observed to have a more crystal-like surface. This indicates that the growth model of the individual nuclei gradually changed from vertical to lateral growth as the C/Si Moore ratio increased. This phenomienon could be explained with the two-dimensional growth model of 3C-SiC islands. The initial stage of carbonization was characterized by different small nuclei of 3C-SiC. With time, 3C-SiC nuclei grow two dimensionally and coalesce, forming larger islands. Then, the island coalescence lead to large platens of 3C-SiC formed on the surface [18]. When the C/Si Moore ratio was less than 1.6, the growth process does not admit the merging of 3C-SiC islands on the surface. According to the reports of Li J.F. [19], due to the large mismatches between SiC and Si, it is reasonable to expect that the SiC nucleation on Si will take either the Volmer-Webber(V-W) mode or Stranski-Krastanov(S-K) mode. It could be shown that the hetero-nucleation of materials with higher surface energy than that of the substrate would result in island formation and subsequent three-dimensional growth, whereas for materials with lower surface free energy than that of the substrate hetero-nucleation would lead to uniform surface coverage and layer-by-layer films growth. According to the view of Zhao Z.F., the high C/Si ratio seem to enhance the arrangement of the well-regulated pattern on Si (100). This means that higher C/Si Moore ratios will decrease the surface free energy [20]. In the Fig. 4 c and d, SEM magnified images showed that these spots mentioned above were some small 3C-SiC grains or preferential oriented columnar 3C-SiC crystals.



a b c d e f

Fig. 4. SEM pictures of 3C-SiC/Si (100) grown at 1385 °C with different C/Si Moore ratios: a – C/Si = 0.8; b – C/Si = 1.0; c – C/Si = 1.2; d – C/Si = 1.4; e – C/Si = 1.6; f – C/Si = 2.0

Fig. 5 shows the surface AFM images of 3C-SiC films gown with different C/Si Moore ratios. Due to the anisotropic growth rates, the 3C-SiC nuclei gradually expand into a pyramidal-like shape with 3C-SiC. Thus, a mosaic-like surface suggested the typical antiphase domain boundaries characteristic of 3C-SiC films [20]. With C/Si Moore ratio increasing from 0.8 to 1.4, 1.6, respectively, the surface roughness of 3C-SiC films decreased from 80 nm to 12 nm, 3.6 nm, respectively. However, after the C/Si Moore ratio increased to 2.0, the surface roughness of 3C-SiC film increased to 4.1 nm. This indicated that the variation of roughness depended on C/Si Moore ratio, significantly. Mirror-like 3C-SiC surface was obtained by growing 3C-SiC film with 1.6 C/Si Moore ratio at 1385 °C.



a b

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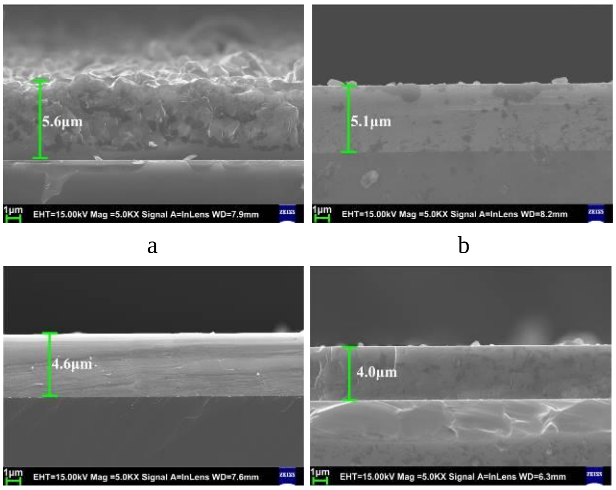
Fig. 5. AFM images of 10 × 10 areas of 3C-SiC/Si(100) growth at 1385 °C with different C/Si Moor ratios: a – C/Si = 0.8; b – C/Si = 1.4; c – C/Si = 1.6; d – C/Si = 2.0

Fig. 6 shows the SEM cross section images of 3C-SiC films, which were grown with different C/Si Moore ratios. With the C/Si Moore ratio increased from 0.8 to 1.4, 1.6, 2.0, respectively, the thickness of the 3C-SiC film decreased from 5.6 μm to 5.1 μm, 4.6 μm, 4.0 μm. It is concluded that the growth rate of the 3C-SiC films depended on the C/Si Moore ratio. The sample grown at 1385 °C with a C/Si Moore ratio of 0.8 showed that the crystallinity was very poor. The surface quality of the 3C-SiC film was a litter unfairness. However, a flat surface could be obtained when the C/Si Moore ratio increased to 1.6.

The crystallinity of 3C-SiC films grown under different C/Si Moore ratios were evaluated by the high resolution X-ray diffraction. Fig. 7 shows the X-ray diffraction patterns. The peak at 69.4° corresponds to the Si (400) planes originating from the substrate. An additional peak at 31.1° appears at the position where the Si (200) peak should appear. The (200) peak is a forbidden reflection, but is very sharp with a broad diffuse region at the base. It has been seen on substrates from various vendors. The appearance of this peak could be due to double diffraction effects. Two typical diffraction peaks located at $2\theta = 41.5°$ and 90.3° correspond to cubic SiC (200) and SiC (400), respectively. Other diffraction peaks located at $2\theta = 35.8°$, 60.2°, 72.08°, and 76.4° corresponding to SiC (111), SiC (220), SiC (311), and SiC (222), respectively. In Fig. 7 a and b, almost whole 3C-SiC peaks were detected, which mean that the 3C-SiC films were polycrystalline layer. Which mean that the vertical growth model caused by the lower C/Si Moore ratio (< 1.6) would lead the 3C-SiC grains growth along the uncertain direction. It is obvious that the XRD diffraction spectrum exhibited quite distinct character as the different C/Si Moore ratios changed. In Fig. 7 c and d, the results showed that the character diffraction spectrum were affected by the different C/Si Moore ratios. When the C/Si Moore ratio was more than 1.6 (including 1.6), only the (200) and (400) 3C-SiC peaks were detected. This indicates that the lateral growth model caused by higher C/Si Moore ratio (≥ 1.6) would form high oriented 3C-SiC films on Si (100) substrates.



a b

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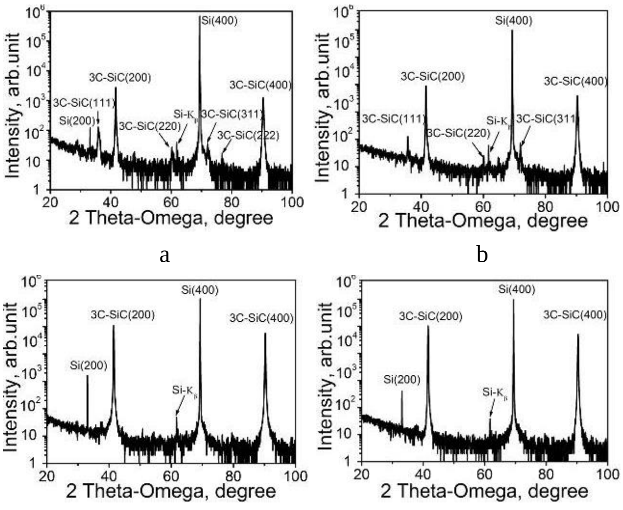
Fig. 6. SEM section pictures of 3C-SiC films grown at two different C/Si Moore ratios: a – C/Si = 0.8; b – C/Si = 1.4; c – C/Si = 1.6; d – C/Si = 2.0



a b

c d

Fig. 7. The 2θ-ω X-ray spectrums of 3C-SiC films grown on the Si (100) substrates at 1385 °C with different C/Si Moore ratios: (a) C/Si=0.8 (b) C/Si=1.4 (c) C/Si=1.6 (d) C/Si=2.0

In Fig. 8, the full width at half maximum (FWHM) values of intense (200) reflection of 3C-SiC films grown with different C/Si Moore ratio by XRD rocking curve scan. Before the C/Si Moore ratio increased to 1.6, the crystalline quality of 3C-SiC films improved with the C/Si Moore ratio increasing. After the C/Si Moore ratio above 1.6, the crystalline quality of 3C-SiC films become a litter worse. It showed that the optimum C/Si Moore ratio for high oriented and high crystal quality 3C-SiC films was 1.6.

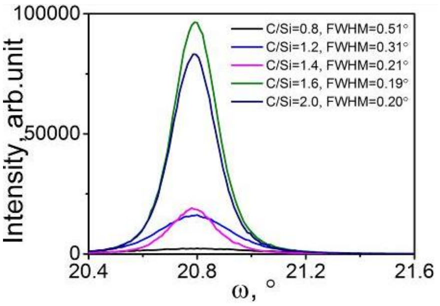


Fig. 8. The rocking curve scans of 3C-SiC films grown on Si (100) at 1385 °C by changing the C/Si Moore ratios from 0.8-3.0

Fig. 9 shows the Raman specta of 3C-SiC/Si (100) films grown at 1385 °C with different C/Si Moore ratios by the modified two-step carbonization method. The band at 940 – 990 cm^{-1} was attributed to the second order of the Si TO-mode related to the Si substrate [21]. The amorphous carbon D and G peaks, respecttively at 1350 cm^{-1} and 1600 cm^{-1}, in the 3C-SiC films were not observed. According to the reports of Wang C. and Teklinska D. [22, 23], there was no carbon species in the 3C-SiC films. Peaks at approximately 794.8 and 969.5 cm^{-1} corresponded to the SiC transverse optical (TO) phonon and longitudinal optical (LO) phonon, respectively. The weak band near 1500 cm^{-1} was attributed to the second order of the 3C-SiC TO-mode. This confirmed that all the SiC film samples were 3C polytype. With the C/Si Moore ratio increased, the shift of the typical TO and LO peaks of 3C-SiC films were hardly observed. The FWHM values of the LO-Raman mode of 3C-SiC films grown with different C/Si Moore ratio were displayed in the Table 2. For the C/Si < 1.6, the FWHM of the LO-Raman mode decreased as the C/Si Moore ratio increased, indicating better layer quality in terms of single crystal growth. According to the reports of Wilhelm M. and Bosi M. [8, 24], the crystal quality of 3C-SiC films could be obtained by comparing the relative intensities of LO and TO peaks of 3C-SiC films. Their studies showed that the crystal quality increased with the increase of LO to TO peak ratio. Table 2 shows the LO to TO peak ratio results. The results show that when the C/Si ratio was less than 1.6, the crystallization quality of 3C-SiC increased as the C/Si Moore ratio increased. After the C/Si Moore ratio exceeds 1.6, the crystallization quality decreased as the C/Si Moore ratio increased. Furthermore, due to the broad band peak was not detected between 750 cm^{-1} to 800 cm^{-1}, it could prove that there was not amprpphous Si-C compound. Based on the results of SEM and XRD, this indicates that a C/Si Moore ratio of 1.6 was the most favorable growth condition for high crystallinity and high morphology 3C-SiC films on Si (100) by the modified two-step carbonization method. The FWHM value of 3C-SiC LO-mode was about 5.0 cm^{-1}, which was close to the best result found in the literature for bulk-grown 3C-SiC [25] (~ 4 cm^{-1}).

Table 2. FWHM Values of LO band and intensity ratios of LO to TO band in 3C-SiC on Si (100) grown under different C/Si Moore ratios

C/Si Moore ratio	FWHM of LO peak, cm^{-1}	LO to TO peak ratio
0.8	35	1.64
1.0	31	2.31
1.2	8.1	2.82
1.4	5.1	9.41
1.6	5.0	17.53
2.0	5.2	15.07
3.0	5.1	12.65

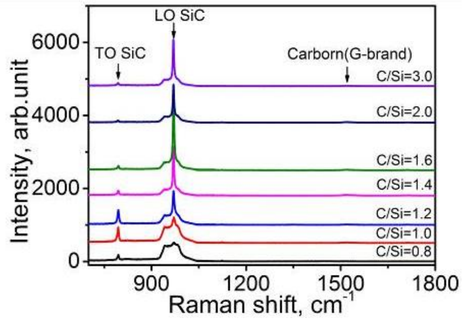


Fig. 9. The Raman spectrums of 3C-SiC/Si (100) grown at 1385 °C by changing the C/Si Moore ratios from 0.8 – 3.0

4. CONCLUSIONS

Through changing the C/Si Moore ratios in the growth procedure from 0.8 – 3.0, the effect of C/Si Moore ratios on the surface quality and crystallinity of 3C-SiC films grown on the Si (100) substrates were studied by the modified two-step carbonization method. As the C/Si Moore ratio increased, the surface morphology of 3C-SiC films changed to smooth mosaic-like surface from rough grain-like surface, significantly. It indicates that the growth model of the individual nuclei gradually changed from vertical to lateral growth as the C/Si ratio increased. The island coalescence lead to large platens of 3C-SiC formed on the surface. According to the results of the SEM cross section images, the growth rate of the 3C-SiC films depended on the C/Si Moore ratio. After the C/Si Moore ratio exceeds 1.6, the crystallization quality decreases as the C/Si Moore ratio increased. The mirro-like 3C-SiC films on the Si (100) was grown with a 1.6 C/Si ratio at 1385 °C for 1hr. The FWHM value of 3C-SiC (200) peak was about 0.19°, which was the smallest in the 3C-SiC films grown with the different C/Si Moore ratio. Therefore, we can conclude that a C/Si Moore ratio of 1.6 is the most favorable growth condition for high crystallinity and morphology 3C-SiC/Si (100) films grown at 1385 °C by the modified two-step carbonization method. The measured surface roughness was 3.6 nm RMS.

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