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To cite this article: Z L Liu et al 2008 J. Phys.: Conf. Ser. 100 042040

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Effect of Si/C flux ratio on the growth of 3C-SiC on Si (111) by SSMBE

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Abstract. The effect of Si/C flux ratio on 3C-SiC grown on Si (111) by solid-source molecular beam epitaxy (SSMBE) is investigated by reflection high energy electron diffraction (RHEED), X-ray diffraction (XRD), atomic force microscopy (AFM) and Fourier transform infrared spectroscopy (FTIR). The results indicate that there is an optimized Si/C flux ratio (1.5:1). In this case, besides SiC streaks, a (3×3) surface reconstruction can be observed in RHEED and the full width at half maximum (FWHM) of the rocking curve is 1.1°. For the sample grown at lower Si/C flux ratio (1.1:1), there are spots as well as ring patterns of SiC observed in RHEED and the FWHM of the rocking curve is 2.1°. For the sample grown at higher Si/C flux ratio (2.3:1), the RHEED indicate that Si spots coexist with SiC spots and the result of the rocking curve shows the FWHM of 1.5°. AFM results show that the surface of the sample grown at the optimized Si/C flux ratio is even and there are voids on the surface of the others. The results of FTIR indicate that the quality of the sample with optimized Si/C flux ratio is best. The more voids and defects of the samples with lower and higher Si/C flux ratios induce larger stress than that of the sample grown at optimized Si/C flux ratio (1.5:1).

1. Introduction
SiC is a wide band gap IV-IV compound semiconductor absorbed more attention due to great technological interest in device operating at high temperature, high power, high frequency and in harsh environment\cite{1,2,3}. Because of the advantages of epitaxy of SiC on Si, many persons try to grow SiC on Si substrate. However, many factors affect the quality of the films. Among them, Si/C flux ratio is thought to be important for the growth of SiC thin films. Many persons investigated the effect of Si/C flux ratio based on CVD\cite{4,5,6,7}. Fewer people studied such effects on the basis of SSMBE. In this paper, the effect of Si/C flux ratio on 3C-SiC film grown on Si(111) by SSMBE is investigated and the optimized Si/C flux ratio is obtained.

2. Experimental
The SiC samples were grown on Si(111) by using the electron gun evaporators for Si and C with base pressure of $5 \times 10^{-8}$Pa in a SSMBE system. During the growth, the samples were characterized by RHEED and the deposition rates of Si and C were measured by quartz crystal oscillators (MAXTEK TM-350) with the precision of 1Å.

Si (111) substrates (n-type) were cleaned for 5min with carbon tetrachloride, acetone and alcohol, then treated with H$_2$SO$_4$:H$_2$O$_2$ (1:1), and dipped in 10% buffer HF solution for 3 min to remove the
After dried in a flux of N₂, the samples were placed into the chamber at once. Afterwards, a Si buffer layer was grown on the substrate with the temperature of 700°C.

The 3C-SiC films were fabricated on Si(111) with the substrate temperature of 1100°C at different Si/C flux ratios: lower (1.1:1), medial (1.5:1), higher (2.3:1) by changing the deposition rate of Si. The growth processes were monitored by RHEED. The crystallinity of films was characterized by XRD spectra obtained using the synchrotron radiation (λ=0.13567nm) at the X-Ray diffraction and scatter station in NSRL of China. The film morphology was measured by AFM performed in tapping mode with a Dimension TM3100 Digital instruments apparatus.

3. Results and discussion

Fig.1 shows the RHEED patterns of a Si buffer layer on Si substrate (A) and the SiC samples with different Si/C flux ratios: (B)(1.1:1), (C) (1.5:1), (D) (2.3:1) respectively. A Si (7×7) reconstruction can be observed in fig.1. (A), which indicates a clean and ordered Si substrate surface. For the sample grown at the lower Si/C flux ratio(1.1:1) as shown in fig.1.(B), besides SiC spots, the faint ring patterns and SiC twin spots are also observed, which indicates that the quality of the sample at lower Si/C flux ratio is worse[3,8]. With the increase of the Si/C flux ratio (1.5:1) as shown in fig.1. (C), we can see the SiC streaks, while SiC twin spots are not observed. This indicates that the surface of the sample is even. A 3×3 surface reconstruction caused by the Si atoms absorbed on the surface of the film was also observed in fig.1. (C). Due to the high substrate temperature, the Si atoms of the substrate diffuse towards the surface. When the sample was cooled down to room temperature from high substrate temperature, the Si atoms of the surface could not be completely evaporated. The SiC (111) - 3×3 surface is terminated by a few consecutive Si atom layers on the outermost layer.[9]For higher Si/C flux ratio (2.3:1) as shown in fig.1.(D), besides SiC spots, some Si faint spots could also be observed, indicating that there are a few Si atom layers on the surface, which were induced by the excess of the evaporated Si atoms. This indicates that the quality of the sample grown with higher Si/C flux ratios is worse[3,8].

![Fig. 1 RHEED patterns of Si substrate (A) and SiC samples with different Si/C ratio (B) (1.1:1) (C) (1.5:1) (D) (2.3:1),](image)

![Fig.2. the rocking curves of SiC(111) diffraction of samples with different Si/C flux ratios(A: 1.1:1, B: 1.5:1, C: 2.3:1)](image)
flux ratio is also worse. According to the result of the RHEED patterns, we conclude that the quality of the sample grown at the medial Si/C flux ratio (1.5:1) is best.

Fig.2. shows the rocking curves of the three samples with different Si/C flux ratios. We can see that the value of FWHM of the rocking curve of the sample grown at the medial Si/C flux ratio is 1.1° and the values of the samples grown at lower and higher ratios are 2.1° and 1.5° respectively. The result also indicates that the quality of the sample grown at medial Si/C flux ratio is best which consists with that of RHEED.

Fig.3. shows the AFM images of SiC samples with different Si/C flux ratios (A: 1.1:1; B: 1.5:1; C: 2.3:1) respectively. There are some voids on the surface of the samples with lower and higher Si/C flux ratios, but less voids on the surface of the sample grown at medial Si/C flux ratio. Compared to fig.3 (A) and fig.3 (C), we can see that the surface roughness of the sample with medial Si/C flux ratio is smaller. The result is consistent with the RHEED observation. A lot of grains are found in fig.3 (A) and (C), which are probably from excess of Si atoms. For the sample grown at the lower Si/C ratio, due to high substrate temperature, the Si atoms of the substrate diffuse toward the surface from the voids. For the sample with higher Si/C flux ratio, the excess of the evaporated Si atoms cause the accumulation of Si atoms on the surface.

In the process of SiC epitaxy on Si surface, due to high substrate temperature, the outdiffusion and evaporation of Si atoms cause the decrease of Si atoms and induce the formation of voids [10, 11, 12, 13]. The voids degrade the crystalline quality, so it is important to suppress or decrease the formation of voids. Our experiment results show that for lower Si/C flux ratio, due to the lack of Si atoms, the outdiffusion of the Si atoms cause more voids. For higher Si/C flux ratio, the large roughness is...
attributed to the excess of evaporated Si atoms. While the optimized Si/C flux ratio can obtain the best quality of the sample. According to the results of AFM, we can see that controlling the Si/C ratio can decrease the voids and improve the quality of the film.

Fig.4. shows the FTIR spectra for the three samples with different Si/C flux ratios (A: 1.1:1, B: 1.5:1, C: 2.3:1). In this figure, for each sample, we can see two sharp peaks that corresponds to the TO and LO phonon mode of 3C-SiC respectively. For the samples with lower and higher Si/C flux ratios, the peaks of LO are close to 950 cm\(^{-1}\) and 953 cm\(^{-1}\) respectively and that of TO are all close to 791.2 cm\(^{-1}\). While for the sample grown at medial Si/C flux ratio (1.5:1), it shows two distinct peaks at 796 cm\(^{-1}\) (TO) and 965 cm\(^{-1}\) (LO). So the value of TO for medial Si/C flux ratio is close to that of TO of the crystal SiC (796.2 cm\(^{-1}\))\(^{[14]}\). Compared to the sample with the medial Si/C flux ratio, the TO peaks of the two others shift toward lower frequency, which is attributed to the stress of the films\(^{[15]}\). Obviously, the more voids and defects of the samples with lower and higher Si/C flux ratios induce larger stress than that of the sample grown at medial Si/C flux ratio. The results of FTIR show that the quality of the sample with medial Si/C flux ratio is best, which is consistent with the observation of RHEED, XRD and AFM.

4. Conclusions
The effect of Si/C flux ratio on 3C-SiC films grown on Si(111) by SSMBE is investigated by using RHEED, XRD, AFM, and FTIR. We find the optimized Si/C flux ratio (1.5:1). The quality of the sample with the optimized Si/C flux ratio (1.5:1) is the best. The results indicate that controlling the Si/C flux ratio can suppress the voids and improve the quality of the SiC film.

Acknowledgement
This work is supported by National Natural Science foundation of China (No.50572100). The authors would like to thank Z.H. SUN and Z.Y. HU for AFM investigations, G.Q. PAN and R.P. LI for XRD measurement and C.X. LI and W. WANG for FTIR.

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