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Growth of void-free 3C-SiC films by modified two-step carbonization methods

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ABSTRACT

Highly preferred orientation 3C-SiC films were deposited on Si (111) substrates by a modified two-step carbonization method. The crystal quality of SiC films were examined by XRD. To evaluate the effect of the introducing of silane during carbonization, cross-sectional SEM was carried out. Results indicated that the introducing of silane during carbonization is effective to prevent the out-diffusion of silicon atoms from silicon substrates. And by adjusting the carbonization conditions, void-free 3C-SiC films were obtained. XPS results demonstrated that the prepared 3C-SiC films were corresponding with the stoichiometric ratio.

1. Introduction

As a wide band-gap semiconductor, cubic silicon carbide (3C-SiC) has attracted a lot of attention for applications in harsh environments due to its superior electrical and chemical properties [1,2]. In addition, 3C-SiC hetero-epitaxial grown on Si substrates is a useful technology for the production of devices such as microelectronics and chemical sensors, and it also can combine the current mature fabrication technology of silicon devices. However, due to large lattice mismatch (20%) and significant difference in thermal expansion coefficients it is difficult to obtain high quality SiC films [3–5]. A common method to solve this problem is to introduce a SiC buffer layer. Usually, this buffer layer was grown by introduce propane under hydrogen flux at lower temperature. Then, the temperature was increased up to the growth temperature and silane was added to grow a thick 3C-SiC layer [6–8]. In addition, until now, many multistep methods were investigated, while, 3C-SiC layer's growth is still characterized by a large number of defects, especially the voids underneath the interface [9–12]. The presence of these voids is attributed to the out-diffusion of silicon atoms from silicon substrates in order to take part to the reaction and create the SiC buffer layer at the initial carbonization stage [13]. In this work, a little silane was introduced during carbonization. The control of the carbonization time and the flux of silane during carbonization were crucial to inhibit the formation of voids between substrates and films. The microstructure of 3C-SiC films and the evolution of the voids in silicon underneath the interface were investigated.

2. Experimental

Experiments were performed in a modified vertical hot wall low-pressure chemical vapor deposition (LPCVD). The susceptor was heated by a graphite heater and the deposition temperature was measured using a W-Re thermocouple located nearly the susceptor and calibrated by an optical pyrometer. Commercially available one face-polished Si (111) wafers with size of 5 × 5 mm^2 were used as substrates. Precursors were SiH_4 and C_3H_8. Purified H_2 was used as carrier gas. The base pressure in reactor chamber before growth was always lower than 10^{-3} Pa. Just before loaded into chamber, the wafers were degreased and dipped in a HF (5%) etching solution for 4 min, then rinsed in deionized water, and flushed in nitrogen flow. Cleaned Si substrates were placed on graphite susceptor in reactor and treated at 1200 °C under hydrogen flow. After this process, temperature was decreased to 900 °C. Then, C_3H_8 was introduced into reactor with a flow rate of 2 sccm (stand cubic centimeter per minute). At the same time the substrates were heated to the growth temperature (1350 °C) within 10 min. This is the first step of carbonization. Subsequently, a little SiH_4 were introduced and growth was maintained 20 min to form the single crystal buffer layer. This is the second modified step of carbonization. To obtain ideal interface, the time for first step of carbonization (T1) and the flux of SiH_4 for second step of carbonization (S2) were varied. The schematic of the modified two-step

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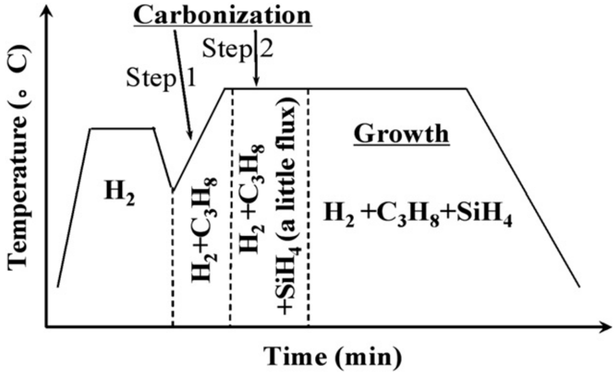

Fig. 1. Schematic showing the modified two-step carbonization methods for the growth of 3C-SiC on Si (111).

Table 1
The carbonization parameters for SiC films.

Samples	Substrate	T1(min)	S2(sccm)
Sample A	Si (111)	5	0.12
Sample B	Si (111)	10	0.12
Sample C	Si (111)	15	0.12
Sample D	Si (111)	15	0.24
Sample E	Si (111)	15	0.36
Sample F	Si (111)	15	0

carbonization method is shown in Fig. 1. The detailed parameters were shown in Table 1. After carbonization, the flux of SiH_4 and C_3H_8 was turned to 100 sccm and 9 sccm, respectively. 60 min later, reactant gases were stopped and substrates were cooled down to room temperature under hydrogen flow.

X-ray diffraction (XRD) of the 3C-SiC films was carried out on an X-ray diffractometer (D-MAX/γA) with $Cu_{K\alpha}$ ($\lambda = 0.154178$ nm) in the range of $30^\circ - 80^\circ$. Rocking curves were measured using X'Pert double crystal diffractometer (Hybrid) with $Cu_{K\alpha1}$ ($\lambda = 0.154051$ nm). Interface morphologies of the SiC films were observed using FE-SEM (JEOL-JSM-6700F). X-ray photoelectron spectra (XPS) were recorded using $Mg_{K\alpha}$ X-ray as the excitation source.

3. Results and discussion

Fig. 2 shows three XRD patterns for 3C-SiC films grown on Si (111) substrates at the same growth conditions with different time for first step of carbonization. When the time for first step of carbonization was 5 min, except two typical diffraction peaks located at $2\theta = 35.6^\circ$ and 75.4° corresponding to cubic SiC (111) and SiC (222) respectively, other two diffraction peak corresponding to SiC (220) and SiC (311) were also observed. It indicated that the obtained SiC film was multi-orientation. As increased T1, the other two diffraction peak corresponding to SiC (220) and SiC (311) disappeared, indicating that the films at least have a preferential orientation which is along to that of Si substrates. Moreover, to assess the crystal quality, X-ray rocking curves of the grown 3C-SiC films were measured. Results are shown in the insets of Fig. 2(a) Fig. 2(b) and Fig. 2(c). From the X-ray rocking curves for SiC (111) it can be seen that the value of full-width at half-maximum (FWHM) for SiC (111) diffraction peak reduced from 0.69° to 0.41° as increase the T1 from 5 min to 15 min. This indicated that 3C-SiC film with high crystallinity has been obtained. While, cross-sectional SEM displays that some voids exist in the interface. It is well known that the presence of these voids is attributed to the out-diffusion of silicon atoms from silicon substrate in order to take part to the reaction [13]. Although in this work, a little silane was introduced in the second modified step of carbonization, while, the voids in interface still exist. It implied that more silane was needed.

XRD patterns for 3C-SiC films grown with different flux of SiH_4 for the second modified step of carbonization were shown in

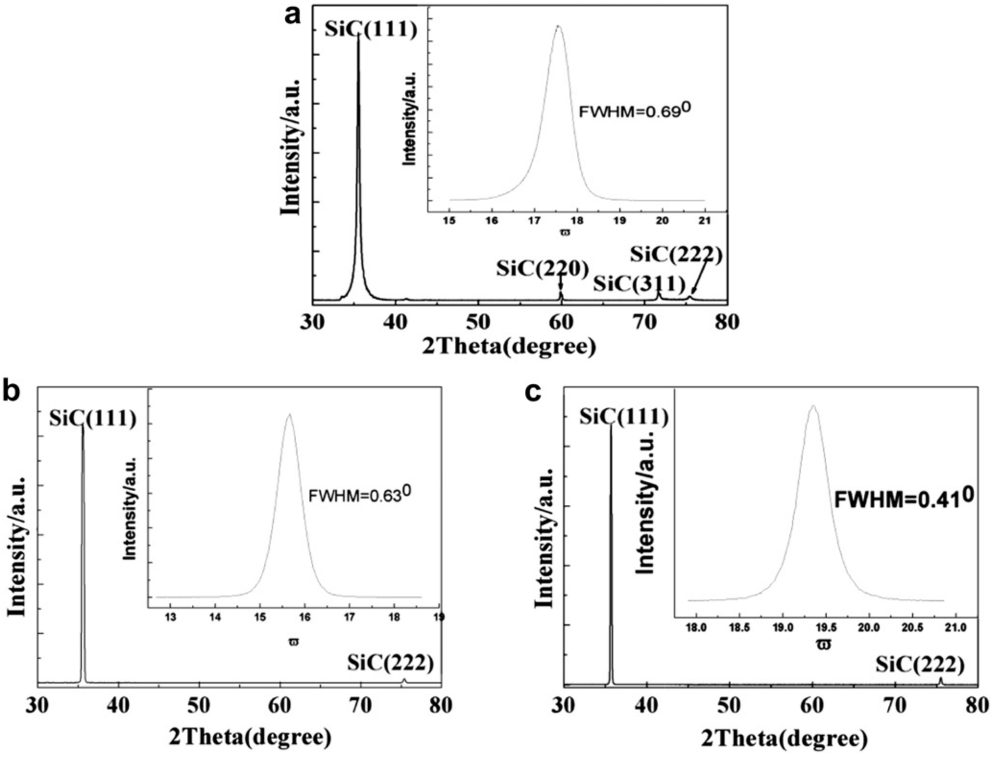

Fig. 2. XRD patterns for SiC films grown with different time for first step of carbonization. (a) T1:5 min; (b) T1:10 min; (c) T1:15 min.