Research Papers

Epitaxial Growth of 3C-SiC on Si(111) Using Tetraethylsilane and Prevention of Void Formation at Heterointerfaces

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The surface morphology and crystalline quality of 3C-SiC films on Si(111) substrates were investigated by using scanning electron microscopy (SEM), transmission electron microscopy (TEM) and Raman spectroscopy, where tetraethylsilane (TES, $\text{Si}(\text{C}_2\text{H}_5)_4$) was first used as a safety source for the growth of SiC films. While TES was expected to be the safest in various organosilane sources, it was essentially anticipated that the high C/Si ratio in TES results in deposition of carbon-rich SiC films, preventing single crystal formation. In raising the temperature after carbonization, voids appeared at the interface of SiC/Si, causing the formation of hillocks on the grown SiC films. The formation of voids and hillocks was prevented by adjusting the TES flow rate and time in the heating process to the growth temperature, leading to the growth of SiC films with a good surface morphology. As a result, it was shown that single crystal epitaxial films could be obtained in the growth of SiC films using TES, and yet a high density of stacking faults resided at the SiC/Si interface.

Key Words : 3C-SiC, MOCVD, Organosilane, SiC/Si Interface Structure, Planar Defects

1. Introduction

Silicon carbide (SiC) thin films have been developed extensively because of their excellent physical and electrical properties, in terms of band gap, breakdown field, saturated electron drift velocity and thermal conductivity, in comparison with Si. The physical and electrical properties of SiC allow for the high-temperature, high-power and high-frequency applications of this material¹⁾.

Chemical vapor deposition (CVD) has been mainly used in growing heteroepitaxial cubic (3C) SiC films on Si, where SiH_4 and C_3H_8 are used as source gases^{2)~4)}. However, strict safety control is required for the use of SiH_4 due to its explosive nature, flammability and toxicity. For SiC growth, the use of organosilane sources such as tetramethylsilane (TMS, $\text{Si}(\text{CH}_3)_4$)^{5)~7)} and hexamethyldisilane (HMDS, $\text{Si}_2(\text{CH}_3)_6$)^{8)~10)} from the viewpoint of environmental safety has been reported. Organosilane can be employed for the efficient growth of SiC thin films because it contains both Si and C atoms. Moreover, organosilane exhibits a less explosive nature than a silane gas source. In particular, tetraethylsilane (TES, $\text{Si}(\text{C}_2\text{H}_5)_4$) is expected to be the safest in various organosilane sources because its boiling point and flashing point are so high as 426 and 299 K, respectively. Moreover, evaporation pressure is 7.5 kPa at 298 K being suitable to control its transfer to a reactor.

However, there are two problems in the CVD of SiC using organosilane sources. One is the fixed C/Si ratio of organosilane sources (TES : C/Si = 8). The high C/Si ratio in TES resulted in the deposition of carbon-rich SiC films, which prevented single-crystal formation^{11)~13)}. The other problem is the formation of voids at the SiC/Si interface and hillocks on the SiC film surface. The carbonization of Si surfaces has been performed to grow high-quality SiC epilayers, but the process forms voids at the SiC/Si interface^{3),14)~17)}. In addition, it has been reported that hillocks are formed on the SiC film surface due to the out-diffusion of Si atoms from the substrate through void defects during the growth of SiC films^{6),7),9)}. This problem is more severe for the CVD of SiC using organosilane sources than for that using silane gas sources, because the out-diffusion of Si atoms is promoted due to the high C/Si ratio of organosilane sources. For these problems, TES has not ever been used in the CVD of SiC thin films.

We have performed the simulation of SiC film growth using organosilane sources¹⁸⁾. Previous experimental results showed that polycrystalline carbon-rich SiC films have been obtained on Si(100) substrates in the growth of SiC films using TES¹¹⁾. In recent experiments, we have been successful in growing single-crystal 3C-SiC films on Si(111) substrates under the optimum growth conditions¹³⁾.

In this paper, we report on characterization of the

crystalline SiC films grown on Si(111) using TES. The surface morphology and microstructure of SiC films are presented in this paper. In addition, the mechanism of void and hillock formation in the films grown using TES is investigated in this study. First, the surface morphology in the initial stage, called carbonization, prior to the growth of SiC films is shown, which is smooth without voids. Second, the characteristics of the SiC films grown using TES are shown, which depended strongly on TES flow rate and heating time after carbonization. Finally, it will be shown that single-crystal SiC films can be obtained on Si substrates even using organosilane source of high C/Si ratio.

2. Experimental

SiC thin films were grown on Si substrates by metal organic chemical vapor deposition (MOCVD) at a low pressure. n-Type Si(111) wafers with on-axis were used as substrates for the growth of SiC films using TES. The Si wafers were cut into a rectangular shape of 15 mm $\times$ 15 mm and were laid on the susceptor. A vertically aligned quartz tube was used as a reactor. The diameter of the quartz tube was 64 mm. The susceptor was made of SiC-coated graphite and heated by RF induction with a power supply operating at 30 kHz. The temperature of the susceptor was measured through the quartz tube by optical pyrometer. The measured temperature was corrected for the emissivity of graphite. H₂ was used as the carrier gas. TES was transferred to the MOCVD reactor by bubbling its container with H₂ at 278 K. The vapor pressure of TES at 278 K was about 0.21 kPa and the purity of TES was 99.9999% (6 N).

The sequences of carbonization and deposition of the films are shown in Fig. 1. The carbonization of Si substrate prior to SiC deposition was performed by introducing C₃H₈ into the reactor. The susceptor temperature was increased from room temperature to 1373 K for 300 s in a mixture of C₃H₈ and H₂ at a pressure of 8.5 kPa, at which the flow rates of C₃H₈ and H₂ were 0.33×10^{-7} m³/s and 0.10×10^{-4} m³/s, respectively. The temperature of 1373 K was maintained for 120 s.

Next, the temperature was once decreased to 1073 K and the C₃H₈ flow rate was kept at 0.33×10^{-7} m³/s to prevent the evaporation of Si atoms from the substrate¹³. As soon as TES was introduced to the reactor, temperature was increased to 1623 K for 60–180 s in a mixture of C₃H₈ and TES. When the temperature reached 1623 K, C₃H₈ flow was stopped.

The H₂ flow rate was fixed at 1.0×10^{-4} m³/s during the growth of SiC films. The TES flow rate was varied from 0.50×10^{-8} m³/s to 0.10×10^{-7} m³/s. The growth rate was 0.11 nm/s at 1623 K at the TES flow rate of 0.50×10^{-8} m³/s. The deposition was completed by turning off the TES flow followed by the cooling of the substrate to room temperature under H₂ flow.

Characterization was carried out for both carbonized buffer layers and deposited SiC films. The surface morphology of the samples was observed by Nomarski optical microscopy and SEM. The thickness of films was measured using a spectroscopic ellipsometer and SEM. The crystallinity of the samples was characterized by reflection high energy electron diffraction (RHEED) and TEM analyses. Raman spectroscopy was used to investigate the existence of excess carbon in the films.

3. Results and Discussion

3. 1 Carbonization process

To form a void-free buffer layer, the carbonization of Si substrates was performed under various conditions. In the previous experiments, it was found that

Fig. 1 SiC growth sequences.

Fig. 2 RHEED pattern and surface morphology of buffer layer formed at 1373 K with a C₃H₈ flow rate of 0.33×10^{-7} m³/s and a H₂ flow rate of 0.10×10^{-4} m³/s.

a high-quality buffer layer can be obtained at 1373 K with a C_3H_8 flow rate of $0.33 \times 10^{-7} \text{ m}^3/\text{s}$ and a H_2 flow rate of $0.10 \times 10^{-4} \text{ m}^3/\text{s}$ ¹³. **Figure 2(a)** shows the RHEED pattern of the buffer layer carbonized under these conditions. The direction of incidence of the electron beam is parallel to the $\langle 011 \rangle$ azimuth of substrate. A fine streaky-spot pattern of SiC was observed in the RHEED pattern. In addition, **Figure 2(b)** shows the surface morphology observed under a Nomarski optical microscope. A smooth surface with no voids was observed in the Nomarski microphotograph. Thus, the use of optimum conditions gives a void-free buffer layer.

Next, the substrate temperature should be increased above 1573 K for the growth of SiC films on the buffer layers. This is because polycrystalline SiC films were deposited below this temperature¹². After carbonization, C_3H_8 flow was turned off and the temperature

was increased from 1373 K to 1603 K for 120 s in H_2 ambient to deposit a SiC layer. **Figure 3** shows the (a) plan-view and (b) cross-sectional SEM images of the sample heated at 1603 K for 120 s in H_2 ambient after carbonization. It can be observed that a polycrystalline buffer layer exists over the triangular voids in the SEM plan-view image, as shown in **Fig. 3(a)**. Moreover, the formation of the voids on the Si side under the buffer layer was observed in the cross-sectional SEM image, as shown in **Fig. 3(b)**. Therefore, it is deduced that the surface morphology and crystallinity of the buffer layer deteriorated with heating in H_2 ambient after carbonization at 1373 K. In addition, voids are formed at the SiC/Si interface by evaporation of Si atoms from the substrate during the heating of the carbonized substrate to 1603 K¹³.

3. 2 Growth with TES

Next, SiC thin films were grown under the sequence

Fig. 3 SEM images of sample heated in H_2 ambient after carbonization.
(a) Plan-view SEM image and (b) cross-sectional SEM image.

Fig. 4 Plan-view SEM images of SiC films grown at various TES flow rates and heating times after carbonization.

(a) $R_{\text{TES}} = 0.50 \times 10^{-8} \text{ m}^3/\text{s}$, $t = 60 \text{ s}$. (b) $R_{\text{TES}} = 0.10 \times 10^{-7} \text{ m}^3/\text{s}$, $t = 60 \text{ s}$. (c) $R_{\text{TES}} = 0.50 \times 10^{-8} \sim 0.10 \times 10^{-7} \text{ m}^3/\text{s}$, $t = 60 \text{ s}$. (d) $R_{\text{TES}} = 0.50 \times 10^{-8} \sim 0.10 \times 10^{-7} \text{ m}^3/\text{s}$, $t = 180 \text{ s}$. (e) Plan-view SEM image from (d) on expanded scale.

shown in Fig. 1, where the effect of TES flow rate and heating time after carbonization was investigated. Figure 4 shows the plan-view SEM images of the SiC films grown at various TES flow rates and heating times after carbonization. The TES flow rates, R_{TES} , are $0.50 \times 10^{-8} \text{ m}^3/\text{s}$ in Fig. 4(a) and $0.10 \times 10^{-7} \text{ m}^3/\text{s}$ in Fig. 4(b). In Fig. 4(c), the TES flow rate was kept at $0.10 \times 10^{-7} \text{ m}^3/\text{s}$ during the heating to the deposition temperature. When the temperature reached 1623 K, the TES flow rate was reduced to $0.50 \times 10^{-8} \text{ m}^3/\text{s}$, and the SiC film was grown at this flow rate. The heating time after carbonization was fixed at 60 s in Fig. 4(a)-(c).

Hillock with triangular pit appears at $R_{\text{TES}} = 0.50 \times 10^{-8} \text{ m}^3/\text{s}$ as shown in Fig. 4(a). On the other hand, a polycrystalline SiC film was formed at $R_{\text{TES}} = 0.10 \times 10^{-7} \text{ m}^3/\text{s}$ as shown in Fig. 4(b). The average grain size was $\sim 100 \text{ nm}$. However, a continuous and smooth surface with small pits was observed, as shown in Fig. 4(c). It shows that a sufficient supply of TES during the heating to the deposition temperature is effective in preventing the formation of hillocks on the SiC films.

In Fig. 4(d), the heating time after carbonization was set to 180 s, where TES flow rate was set at $0.10 \times 10^{-7} \text{ m}^3/\text{s}$ during the heating, and at $0.50 \times 10^{-8} \text{ m}^3/\text{s}$ during the SiC film growth. In comparison with Fig. 4(c), the size and density of the pits on the surface of SiC film are reduced significantly by setting the heating time to 180 s. It shows that the prolongation of

heating time after carbonization is efficient to prevent the formation of pits on the SiC films. In addition, many triangular features were observed in the plan-view SEM image shown in Fig. 4(d). At higher magnification, Fig. 4(e), the direction along the side of triangle was parallel to the $\langle 011 \rangle$ azimuth^{15),16)}. Such surface morphology probably originates from double positioning boundaries^{19)~21)} or stacking faults as described in TEM characterization.

Figure 5 shows the cross-sectional SEM images of the SiC films grown at various TES flow rates and heating times after carbonization. The thickness of the SiC films is $0.4 \mu\text{m}$. Although a high density of large voids was observed at the SiC/Si interface for the growth at $R_{\text{TES}} = 0.50 \times 10^{-8} \text{ m}^3/\text{s}$ as shown in Fig. 5(a), a void-free interface of the SiC/Si structure was observed at $R_{\text{TES}} = 0.10 \times 10^{-7} \text{ m}^3/\text{s}$ as shown in Fig. 5(b). In addition, the size and density of the voids at the SiC/Si interface were reduced significantly by setting R_{TES} to $0.10 \times 10^{-7} \text{ m}^3/\text{s}$ during the heating as shown in Fig. 5(c) and Fig. 5(d). In particular, the voids were scarcely observed at the SiC/Si interface for the heating time of 180 s as shown in Fig. 5(d). This confirms that void formation at the SiC/Si interface is suppressed by adjusting the TES flow rate and time in the heating process after carbonization. Similar results have been also reported for growth using TMS^{6),7)} and HMDS⁹⁾.

It has been generally reported that the residence of voids at the SiC/Si interface causes the diffusion of Si

Fig. 5 Cross-sectional SEM images of SiC films grown at various TES flow rates and heating times after carbonization.

(a) $R_{\text{TES}} = 0.50 \times 10^{-8} \text{ m}^3/\text{s}$, $t = 60 \text{ s}$. (b) $R_{\text{TES}} = 0.10 \times 10^{-7} \text{ m}^3/\text{s}$, $t = 60 \text{ s}$. (c) $R_{\text{TES}} = 0.50 \times 10^{-8} \sim 0.10 \times 10^{-7} \text{ m}^3/\text{s}$, $t = 60 \text{ s}$. (d) $R_{\text{TES}} = 0.50 \times 10^{-8} \sim 0.10 \times 10^{-7} \text{ m}^3/\text{s}$, $t = 180 \text{ s}$.

atoms from the substrates to the grown layers^{(6),(7),(9)}. In this experiment, voids at the SiC/Si interface were generated during the heating of the carbonized substrate to the deposition temperature. It was mentioned that Si atoms in the substrate diffuse through voids during the SiC film growth reacting with the excess carbon supplied from organosilane source. As a result, hillocks are formed on the surface of the SiC film. In the experimental results, an increase in TES flow rate during the heating to the growth temperature suppresses void formation at the SiC/Si interface. In addition, prolongation of heating time to the growth temperature gives enough time to cover the carbonized surface with the SiC layer at the initial stage of the growth. This results in suppressing the formation of voids at the SiC/Si interface, leading to the growth of hillock-free SiC films.

Fig. 6 Raman spectra of SiC films grown at various TES flow rates and heating times after carbonization.

(a) $R_{\text{TES}} = 0.50 \times 10^{-8} \text{ m}^3/\text{s}$, $t = 60 \text{ s}$. (b) $R_{\text{TES}} = 0.10 \times 10^{-7} \text{ m}^3/\text{s}$, $t = 60 \text{ s}$. (c) $R_{\text{TES}} = 0.50 \times 10^{-8} \sim 0.10 \times 10^{-7} \text{ m}^3/\text{s}$, $t = 60 \text{ s}$. (d) $R_{\text{TES}} = 0.50 \times 10^{-8} \sim 0.10 \times 10^{-7} \text{ m}^3/\text{s}$, $t = 180 \text{ s}$.

Figure 6 shows the Raman spectra of the films grown at various TES flow rates and heating times after carbonization. The Raman spectra show transverse (TO) and longitudinal optical (LO) phonons at 791 and 970 cm^{-1} , respectively, indicating that 3C-SiC films are grown on Si substrates. However, for the SiC film grown at $R_{\text{TES}} = 0.10 \times 10^{-7} \text{ m}^3/\text{s}$, graphite-related peaks were also observed at 1350-1600 cm^{-1} , indicating the incorporation of excess carbon. These peaks disappeared with the decrease in R_{TES} to $0.50 \times 10^{-8} \text{ m}^3/\text{s}$. These results indicate that the degradation of the crystallinity of the SiC film at $R_{\text{TES}} = 0.10 \times 10^{-7} \text{ m}^3/\text{s}$ is due to the formation of a graphite structure in the film. Thus, it is concluded that TES flow rate should be varied during heating to the growth temperature and the deposition of the film to obtain high-quality SiC films. In the experimental results, an increase in TES flow rate during the heating to the growth temperature suppresses void formation at the SiC/Si interface. Moreover, a decrease in TES flow rate during the SiC film growth prevents excess carbon incorporation in the film.

Next, TEM observation was carried out to investigate the crystalline quality and microstructure of SiC films. **Figure 7** shows a TEM plan view image and selected area diffraction (SAD) pattern for the SiC film grown by setting the heating time to 180 s after carbonization [Fig. 4(d)]. The electron beam direction is parallel to [011] azimuth. The SAD pattern taken in the region of $\sim 400 \text{ nm}$ showed one set of strong diffraction spots as shown in Fig. 7(b) indicating single crystal growth. However, the streaks along $\langle 111 \rangle$ spots were observed in the SAD pattern indicating the existence of a high density of stacking faults in the film. This is also confirmed in the TEM plan view image as shown in Fig. 7(a). The sharp linear contrast corresponds to the planar defects lying on the $\{111\}$ planes oriented vertical to the $\{011\}$ planes. In addition, the vague contrast corresponds to the planar defects on the $\{111\}$ planes oriented with 35.3° to the

Fig. 7 TEM microphotographs of the SiC film grown by setting the heating time to 180 s after carbonization.

(a) TEM plan view image, and (b) SAD pattern taken in the region of $\sim 400 \text{ nm}$. The electron beam direction is parallel to [011] azimuth.

Fig. 8 TEM microphotographs at the SiC/Si interface.

(a) Cross-sectional TEM image, and (b) SAD pattern. The electron beam direction is parallel to $[011]$ azimuth.

$\{011\}$ planes. In fact, SAD patterns from many different regions were taken to confirm the monocrystallinity of the SiC over a wide area. Each SAD pattern exhibited the same strong spot pattern. Therefore, it is deduced that the film grown using TES is single crystal 3C-SiC with a high density of stacking faults.

Next, cross-sectional TEM observation was carried out to investigate the microstructure at the SiC/Si interface. Figure 8 shows a cross-sectional TEM image and SAD pattern at the SiC/Si interface of the same film. In the SAD pattern, it consists of two sets of spots as shown in Fig. 8 (b). The spots with shorter reciprocal lattice vectors represent the Si diffraction pattern, while corresponding outer spots represent the SiC diffraction pattern. The two sets of spots are exactly collinear; i.e. the two crystals have a parallel epitaxial orientation relationship of $[011]_{\text{Si}}//[011]_{\text{SiC}}$ and $(111)_{\text{Si}}//(111)_{\text{SiC}}$. In addition, the streaks associated with the SiC spots were observed in the SAD pattern, which is shown the existence of a high density of stacking faults or twin bands. Similar patterns have been also reported for growth using HMDS^(9),10). Fig. 8(a) shows the cross-sectional TEM image at the SiC/Si interface. Planar defects on the $\{111\}$ plane of SiC film are introduced to accommodate the large lattice mismatch between 3C-SiC and Si substrate. Moreover, a dark contrast is observed in the near region to the Si substrate due to the misfit strain between the SiC film and the Si substrate. As the SiC film grows thicker, the dark contrast becomes lighter for the modification of the misfit strain. The observed results show that 3C-SiC epitaxial film with a high density of stacking faults or twins is formed on the Si(111) substrate.

4. Conclusion

We investigated the characteristics of SiC films on Si(111) using TES as safety sources. Surface morphology and crystalline quality of the films were characterized by SEM, TEM and Raman spectroscopy, and was found to be degraded by both the formation of voids on the substrates and the incorpo-

ration of excess carbon during the growth. For the use of TES as a safe organosilane source, carbon incorporation should be prevented by decreasing the TES flow rate. The formation of voids was suppressed by adjusting the TES flow rate and time in the heating process to the growth temperature, resulting in the growth of SiC films with smooth surfaces. TEM observation revealed that 3C-SiC epitaxial film with a high density of stacking faults and twins could be obtained in the growth of SiC films using TES for the first time. TES is regarded as appropriate source for 3C-SiC growth to provide a safe and simple process compared with conventional silane sources.

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