GROWTH MECHANISM OF 3C-SiC(111) ON Si WITHOUT CARBONIZATION PROCESS

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Abstract – We have used rapid thermal chemical vapor deposition (RTCVD) technique to grow epitaxial SiC thin films on Si wafers without carbonization process by pyrolyzing tetramethylsilane (TMS). The growth rate of SiC films increases with TMS flow rate and temperature, but it decreases with temperature at higher TMS flow rates. The XRD spectra of the films indicate that the growth direction is along the (111) direction of β-SiC. IR and RBS measurements have been employed to analyze the chemical composition of the films. At 1100°C TMS molecules dissociate almost completely into Si atoms, CH_4 and C_2H_2 gases. The growth mechanism of SiC films on Si substrates without carbonization process has been proposed based on the analyses by TEM and QMS.

INTRODUCTION

Silicon carbide (SiC) has currently received much interest as an attractive candidate for electronic devices operating at extreme conditions and blue-LED's, owing to its wide band gap, high breakdown voltage, high thermal conductivity, high saturated electron velocity and good chemical stability [Powell and Will, 1989; Davis et al., 1991; Schlichting, 1980]. Most of the previous works have investigated the growth of SiC thin film on a carbonized Si substrate utilizing chemical vapor deposition with separate C- and Si-bearing precursors, normally requiring high temperatures above 1300°C [Nishino et al., 1987; Steckl and Li, 1992; Avigal et al., 1974; Jacobson, 1971]. In recent years, use of some organosilane as a single precursor has tried for the growth of SiC film at low temperatures below 1000°C [Takahashi et al., 1992; Figueras et al., 1991]. In spite of the success of the SiC film growth on Si with methylsilane at 750°C [Golecki et al., 1992], this technique is not widely used in semiconductor industries because the carbonization process which is neccessary before the growth needs the temperature more than 1300°C. In this work, therefore, we have studied the possibility of high quality SiC growth without carbonization process and the growth mechanism of SiC thin film.

EXPERIMENTAL

The RTCVD reactor, shown in Fig. 1, was specially designed and fabricated for the growth of SiC thin films. High-power tungsten halogen lamps were used to heat a Si substrate. In order to maximize the heating effects, the light from the lamps was focused on the Si substrate in the reactor using specular surfaces set up on the backside of the lamps. The temperature of the substrate was monitored using a Pt/Ru thermocouple

directly inserted in the substrate holder and maintained at a constant value with a temperature controller.

TMS and hydrogen gases were introduced into the reactor through mass flow controller (MFC). TMS reservoir was immersed in icy water bath. Rotary vacuum pump was used to evacuate the reactor up to 10^{-4} Torr. The pressure of the deposition reactor was measured with a convectron gauge. P-type Si(111) wafers with the resistivity of 20 $\Omega \cdot cm$ were used as substrates for the SiC thin film growth. The Si substrate had been cleaned by dipping in acetone and then in HF aqueous solution, and dried with a nitrogen flow, prior to being loaded into the reactor. After mounting the cleaned Si substrate on the substrate holder, the reactor was evacuated up to 10^{-4} Torr and purged with H_2 gas of 20 sccm. Then the reactor was heated up to 1100°C with the flow of H_2 gas, and maintained under the condition for 2 minutes. After the substrate temperature was cooled down to room temperature, the hydrogen flow was discontinued. This process was repeated more than five times to remove completely impurities remaining on the Si substrate. When quadrupole mass spectrometer (QMS) analysis shows that water related peaks go down below the detectable limits, the growth reaction was started.

The reactor was again heated up to the growth temperature at the rate of 20°C/s under the flow of hydrogen gas. Then TMS diluted with H_2 was introduced into the reactor at the experimental flow rate. Experiments for SiC deposition had been carried out for 15 minutes at various substrate temperatures (1000-1100°C), H_2 flow rates (20-200 sccm), and TMS flow rates (0.1-5 sccm). To cease SiC growth, TMS flow was first stopped and then the temperature was dropped to room temperature under the hydrogen flow. The reactor pressure was elevated to the atmospheric pressure by flushing with Ar gas, and the sample was taken out of the reactor for characterization.

The thickness of grown SiC films has been measured with an α-stepper and transmission electron microscopy (TEM). The

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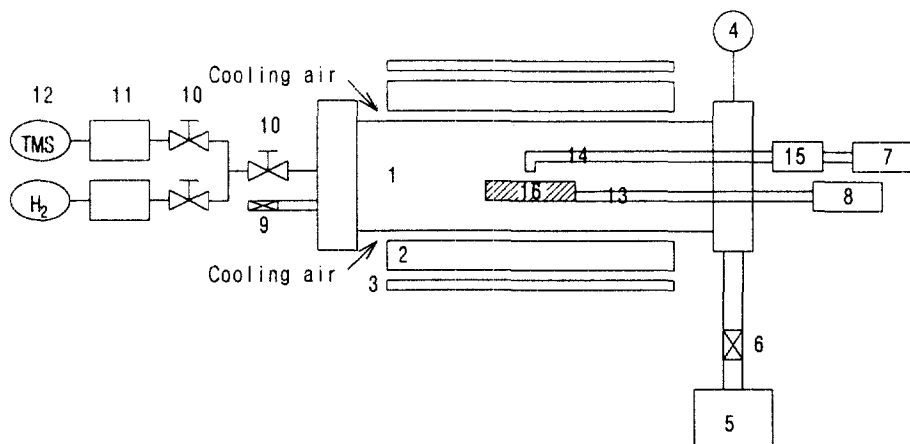†To whom all correspondences should be addressed.

Fig. 1. A schematic diagram of rapid thermal CVD apparatus.

1. Quartz chamber	5. Rotary pump	9. Leak valve	13. Thermocouple
2. Tungsten halogen lamp	6. Gate valve	10. Throttle valve	14. Quartz tube
3. Reflector	7. Mass-spectrometer	11. MFC	15. Filter
4. Vacuum gauge	8. Temp. controller	12. Source gas	16. Substrate

surface morphology of the grown SiC films has been observed with scanning electron microscopy (SEM). X-ray diffraction (XRD) and Raman spectroscopy have been used for the analysis of the orientation of the films. The structure of the films has been determined with TEM and transmission electron diffraction (TED). Infrared spectroscopy (IR) and Rutherford backscattering spectroscopy (RBS) measurements have been employed to analyze the chemical composition of the grown films. During the growth, the gas in the chamber has been sampled using a quartz tube with the diameter of 1 mm, placed 1 mm above the Si substrates, and analyzed with QMS (Hiden, HAL/3F 501).

RESULTS AND DISCUSSION

1. Effects of TMS Flow Rate and Substrate Temperature

The growth rate of SiC thin film was measured at 1100°C and 20 sccm H_2 with varying TMS flow rate. Fig. 2(a) shows that the growth rate increases with the TMS flow rate, which is considered to be due to the increase of reactive species for the SiC growth. This will be discussed in more detail later. QMS analysis of the gases in the reactor during the SiC growth showed that the reactive species increased with the TMS flow rate. Fig. 2(b) shows the growth rate of SiC thin film as a function of substrate temperature at the TMS flow rates of 0.5 and 5.0 sccm. With increasing temperature, the growth rate increases for 0.5 sccm, but decreases for 5.0 sccm.

The decrease of the growth rate at high TMS flow rate is not reported for cold-wall reactor, but is believed to be caused by the properties of the CVD reactor used in this work. The Si substrate is heated by the light emitted from lamps. The light also warms the reactor wall to bring about reaction between the reactor wall and TMS at high TMS concentration, resulting in coating the inside wall of the reactor by black carbon films. This black film absorbs the light more efficiently, and the reactor becomes hot-wall type. It is generally reported that, for hot wall type reactors, the reaction at the reactor wall is accelerated
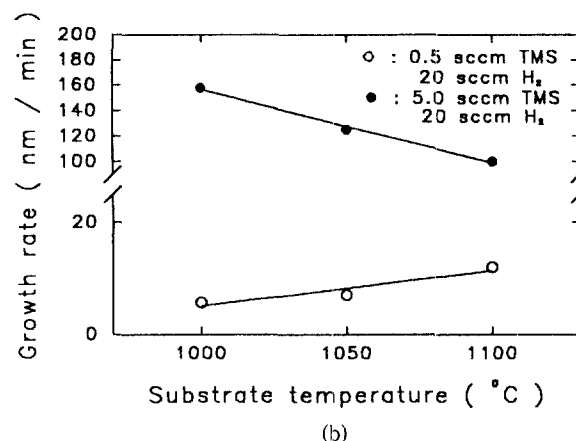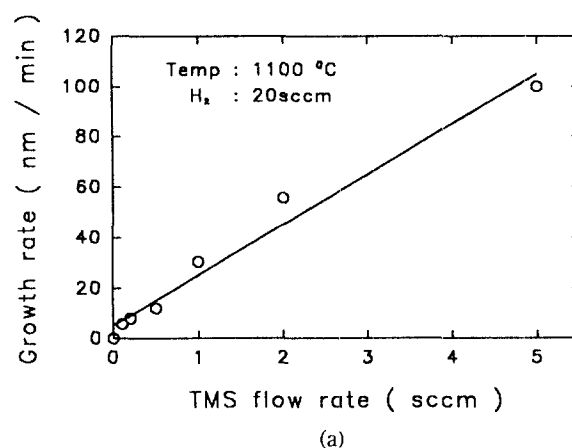

(a)

(b)

Fig. 2. The growth rates of SiC thin film as functions of (a) TMS flow rates and (b) substrate temperature.

with the increase of temperature to deplete reactive species on the surface of the Si substrate, leading to the reduction of SiC growth rate [Chiu et al., 1993].
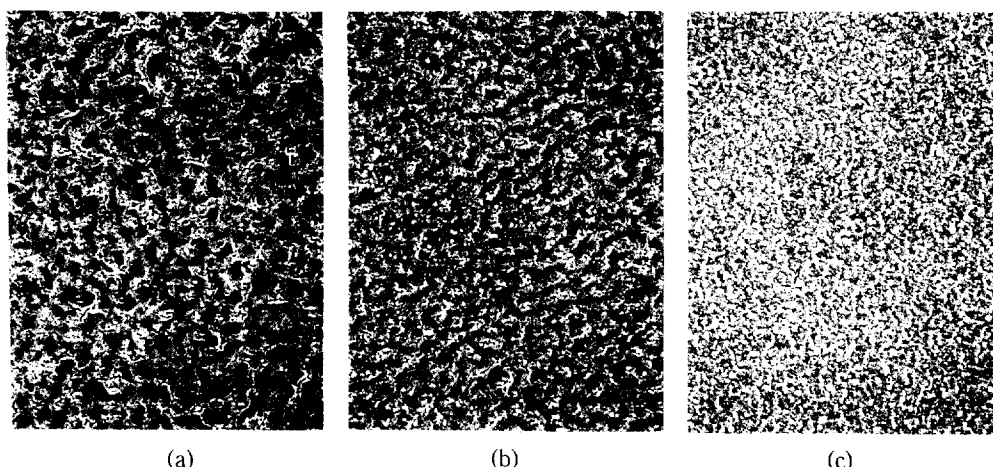
(a)   (b)   (c)

Fig. 3. Scanning electron micrographs of 3C-SiC thin films grown for 15 min at 1050°C and 20 sccm H₂ as a function of TMS flow rate.
(a) 0.2 sccm, (b) 0.5 sccm and (c) 1 sccm.

2. Analyses of SiC Films Grown

In Fig. 3 are shown plane-view SEM micrographs of grown SiC thin films. They are grown for 15 min at 1050°C and 20 sccm H_2 at various TMS flow rates. SiC thin films become denser with increasing TMS flow rate. XRD was used to investigate the orientation of the SiC films grown under experimental conditions included in Fig. 2, and the typical XRD spectrum is shown in Fig. 4(a). It is to be noted from the figure that the films are oriented along the (111) plane of β-SiC. This is also confirmed from the typical Raman spectrum for the films, as shown in Fig. 4(b), which exhibits TO and LO modes of β-SiC(111) at 795 and 972 RCM⁻¹, respectively. Free Si and (200), (220), and (311) planes of β-SiC, which had been frequently obtained during the deposition of SiC thin films using organosilane/H_2 mixture below 1400°C, are not detected. No SiC thin films were deposited on Si substrates below 1000°C. This is believed because the degree of TMS decomposition is very low below 1000°C.

TEM and TED measurements were performed to investigate the structure of the SiC films grown at the pressures of 1.7 and 12 Torr for 15 min at 1100°C when the flow rates of TMS and H_2 were maintained at constant values of 0.5 and 100 sccm, respectively. At 1.7 Torr, a polycrystal SiC film was grown on a Si substrate, and large voids were observed in the Si side of the SiC/Si interface [see Fig. 5(a) and (b)]. This void formation may be due to the out-diffusion of Si atoms, as reported in literature [Mogab and Leamy, 1974]. The same results were obtained from the cross sectional TEM micrographs of the SiC films grown at the H_2 flow rate of 20 sccm at various TMS flow rates between 0.2 and 5.0 sccm and at the substrate temperatures between 1000 and 1100°C, while maintaining the reactor pressure at a low value of about 2.5 Torr. This indicates that polycrystal SiC films are grown at such a low pressure regardless of the TMS flow rates. On the contrary, at 12 Torr, an intermediate SiC layer of 18 nm thick was first grown on the Si substrate, and then a single-crystal SiC thin film with many twins and defects grows on the layer [see Fig. 5(c) and (d)]. Voids were also formed in the Si side of the SiC/Si in-
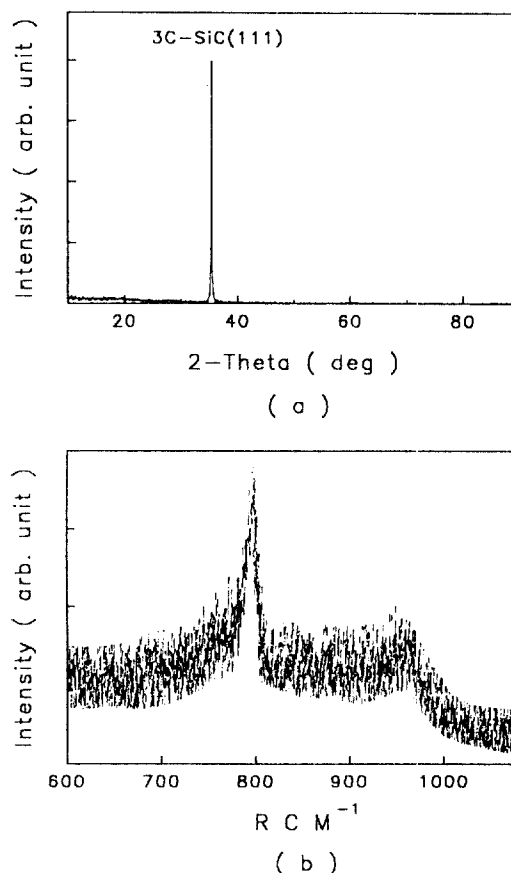

Fig. 4. (a) Typical XRD pattern and (b) Raman spectrum of 3C-SiC thin films grown for 15 min at 20 sccm H₂ and 0.5 sccm TMS.

terface. As the total pressure of the reactor increases, the thickness of the grown SiC film was reduced from 371 to 171 nm and the size of the voids formed in the Si substrate decreased. IR was used to analyze the chemical composition of the SiC films grown at 20 sccm H_2 and 0.5 sccm TMS at various sub-
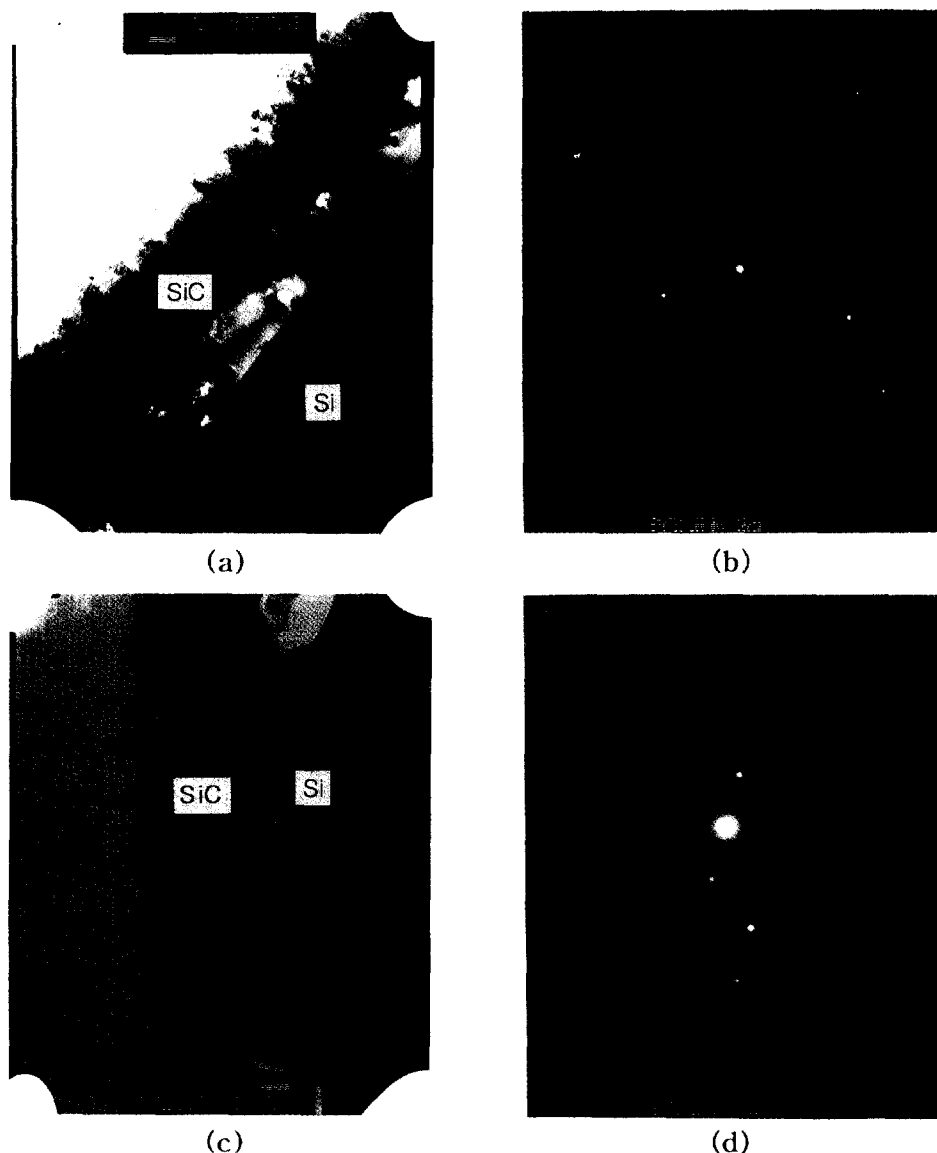
(a)

(b)

(c)

(d)

Fig. 5. Cross-sectional TEM micrographs and diffraction patterns of 3C-SiC thin films grown for 15 min at 1100°C, 20 sccm H_2 and 0.5 sccm TMS as a function of total gas pressure.
(a) and (b) 1.7 Torr, and (c) and (d) 12 Torr.

strate temperatures. Fig. 6 shows that a strong peak of Si-C stretching mode appears at 800 cm⁻¹ and the absorption intensity of the peak increases very rapidly with increasing temperature. Si-Hn at ~2100 cm⁻¹, Si-CH₃ at ~1010 cm⁻¹, and C-H at 2800-3000 cm⁻¹ were not observed in the films. RBS analysis shows that the SiC films are stoichiometric (see Fig. 7).

3. Analysis of Reaction Gas

To investigate the major reactive species for the SiC deposition, gaseous chemical species generated by thermal cracking of TMS in the reactor were analyzed by QMS. It is well known in QMS measurements that self-cracking also occurs in QMS even if the gas is not intentionally cracked [Motoyama, 1990]. In order to remove the effects of self-cracking, measurements were performed both at noncracking (25°C) and cracking temperature (1100°C), and the measured peaks were normalized with the intensity of m/e=73 [$(CH_3)_3Si$] at 25°C. The

results of QMS measurements for TMS are shown in Figs. 8(a) and (b) for the cases of noncracking and cracking conditions, respectively. Fig. 8(a) shows the self-cracking pattern of TMS in QMS, in which the peak intensity of each species is normalized with that of $(CH_3)_3Si$. The flow rates of H_2 and TMS were 20 and 0.5 sccm, respectively, and the total pressure was 2.5 Torr. As shown in the figure, CH_3SiH_n (m/e=43-45) and $(CH_3)_3Si$ (m/e=~73) are the major chemical species produced by the self-cracking of TMS in QMS at 25°C. CH_n, C_2H_n, SiH_n, and $(CH_3)_2Si$ also appear with weak intensities at m/e of ~14, ~26, ~30, and ~58, respectively. The result of QMS measurements for TMS at 25°C is in good agreement with those of the previous report [Stenhagen et al., 1969]. Fig. 8(b) indicates the cracking pattern of TMS at 1100°C. The peak intensity of each species is normalized with that at 25°C. Comparing Figs. 8(a) and (b), remarkable distinctions can be recognized. The
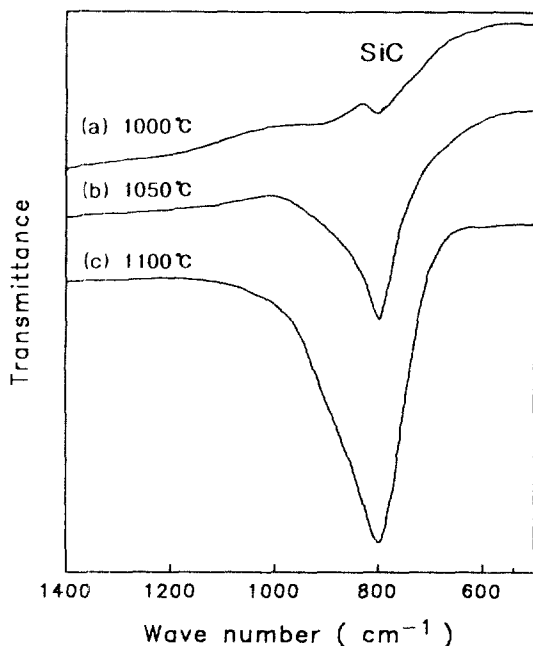
Fig. 6. IR spectra of 3C-SiC thin films grown for 15 min at 20 sccm H₂ and 0.5 sccm TMS as a function of substrate temperature.
(a) 1000°C, (b) 1050°C and (c) 1100°C.

Fig. 8. Quadrupole mass spectra for TMS diluted with hydrogen
(a) self-cracking pattern in QMS at 25°C. (b) thermal cracking pattern at 1100°C and 0.5 sccm TMS. (c) thermal cracking pattern at 1100°C and 5 sccm TMS.
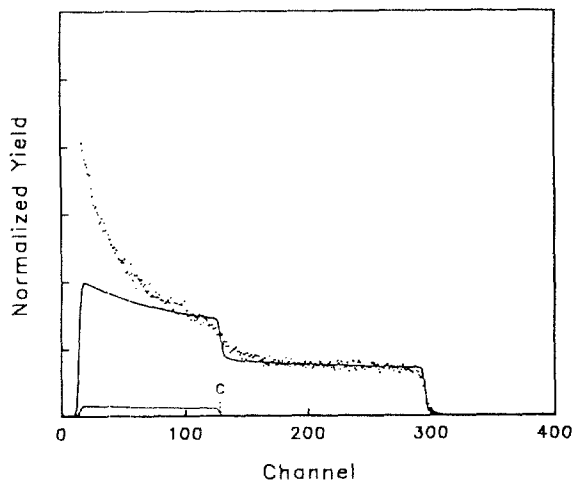

Fig. 7. RBS spectrum of 3C-SiC thin films grown for 15 min at 20 sccm H₂ and 0.5 sccm TMS.

peaks related to species containing Si [$(CH_3)_3Si$, $(CH_3)_2Si$, CH_3Si, and SiH_n] disappear almost completely at 1100°C. Avigal and Schieber [1974] reported that TMS decomposes into SiH_n, CH_3, and hydrogen at the SiC growth temperature, and SiH_n in turn dissociates at this temperature to form a solid Si and hydrogen. In this experiment, however, the peaks related to SiH_n disappears completely at 1100°C. This is because SiH_n which is generated by the thermal cracking of TMS has too short life time at the growth temperature to be detected by QMS. It had been reported that SiH_n is completely disintegrated into Si and hydrogen at above 900°C [Maya, 1994; Veintermillas et al., 1995] carried out a thermodynamic calculation of the thermal decom-
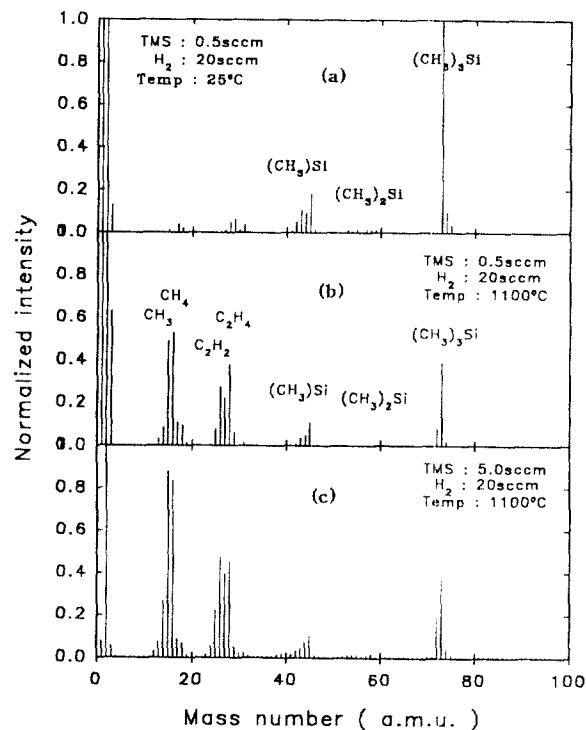
position of TMS under the equilibrium condition, and insisted that the concentration of gaseous Si atoms increases with TMS pressure, but the free Si atoms are completely depleted when the SiC growth process takes place. Gas-phase Si atoms have also been observed during the thermal cracking of organosilanes by various analytic techniques [Ho and Breiland, 1988].

Fig. 8(b) also shows that the peaks around m/e=15 and 16, and 25 and 26, which originate from CH_4 and C_2H_2, respectively, increase remarkably at 1100°C. The peaks related to C_2H_4 (m/e=27, 28) also increases. Herlin et al. [1992] observed in their experiments that the thermal decomposition of TMS generates deposition of carbon and silicon atoms, and a gas phase consisting of H_2, CH_4, C_2H_2, and C_2H_4. They also found that the composition of the gas phase is similar to the products from pyrolysis of ethane (C_2H_6). Based on these analyses, they proposed the mechanism of TMS decomposition as follows: CH_3 radicals generated by thermal decomposition of TMS might be united to form ethane but only momentarily, since ethane decomposes rapidly into H_2, CH_4, C_2H_2, and C_2H_4 at this temperature. From our observations, it is noteworthy that C_2H_6 generated by the reaction of two methyl radicals is not detected by QMS because it decomposes very rapidly at 1100°C to form mainly CH_4 and C_2H_2.

The QMS spectra of TMS, obtained at the same condition as in Fig. 8(b) except that the flow rate of TMS is 5 sccm, are shown in Fig. 8(c). It is seen that the tendency of the spectrum coincides well with that of Fig. 8(b), but the intensities of the peaks related to hydrocarbon compounds such as CH_4 and C_2H_2

increase substantially. This indicates that the amounts of gaseous Si atoms and hydrocarbon compounds increase with increasing TMS flow rate. Summarizing QMS analyses, TMS is thermally decomposed at the SiC growth temperature to generate gaseous Si atoms and CH_3 radicals. Two methyl radicals are then united to form C_2H_6. However, this formed C_2H_6 decomposes rapidly at 1100°C to produce mainly CH_4 and C_2H_2.

4. Growth Mechanism

According to the analyses using QMS, TMS breaks thermally into gaseous Si atoms and CH_3 radicals. CH_3 radicals form C_2H_6 and then quickly breaks into CH_4, C_2H_2, C_2H_4 and hydrogen. TEM analyses revealed that voids are formed in the Si substrate regardless of experimental conditions, though their sizes are different. Also it was revealed that the intermediate SiC layers above the void are reduced to 18 nm thick when the total pressure increases. These experimental results suggest that the growth reaction occurs in two steps when SiC layer is grown on Si substrates using TMS/H_2 without carbonization
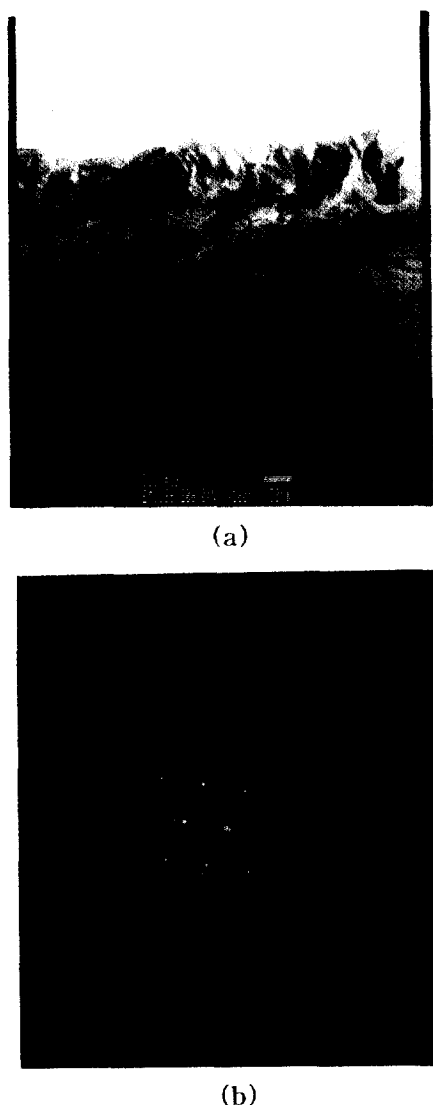
(a)

(b)

Fig. 9. (a) Cross-sectional TEM micrographs and (b) diffraction pattern of 3C-SiC thin film obtained on the carbonized surface of silicon substrate.

process: (1) Si atoms on the surface of substrates react with C-containing gases generated by thermal cracking of TMS and form a C-terminated carbonized layer, and (2) gaseous Si atoms and C-containing gases alternatively reacts to form a single crystalline β-SiC layer on top of the carbonized layer.

Density of C atoms is 4 times higher than that of Si atoms in the reaction chamber since TMS breaks into 1 gaseous Si atom and 4 CH_3 radicals. These abundant CH_3 radicals accelerate the carbonization of surface Si atoms, leading to the depletion of Si atoms from the substrate surface. This depletion causes the outdiffuion of Si atoms from the inside of Si substrate to the surface to maintain stoichiometry as observed by RBS measurements because Si atoms supplied from the cracked TMS are small. The outdiffused Si atoms further react with adsorbed hydrocarbon radicals to form an intermediate SiC layers. When the gas pressure of the reactor is low during this process, the outdiffusion rate of Si atoms is fast due to the high vapor pressure of silicon at low pressure, and the growth reaction at the surface is fast, resulting in the formation of thick intermediate SiC layer. But the crystallinity of thus formed SiC layer is bad, since supply rates of Si and C atoms are higher than the migration rates of the atoms on the surface. On the other hand, when the gas pressure of the reaction chamber is high, outdiffusion of Si atoms from the bulk of substrates is suppressed and hence even thin intermediate SiC films prevent the outdiffusion of Si atoms from the substrate. Therefore, the higher the gas pressure of the chamber, the thinner the intermediate SiC layer formed over voids.

Once the intermediate SiC layer becomes thick enough for a given gas pressure to hinder the outdiffusion of Si atoms from the Si substrate, as explained above, the main source of Si atoms needed for the SiC growth reaction is gaseous Si atoms generated by the decomposition of TMS. The gaseous Si atoms adsorb on the C-terminated surface of the intermediate SiC layer and react to form Si-terminated surface. However, the reaction rate is very slow due to the small amount of Si atoms supplied from thermally cracked TMS. Then C-containing gas, for example C_2H_2, adsorbs on the Si-terminated surface and dissociates into C atoms and hydrogen gas. the formed C atoms thus react with surface Si atoms to form C-terminated surface and the reaction continues. In this way, a single crystalline β-SiC layer is grown on the intermediate layer since the amount of Si atoms supplied from TMS is small and then migration times of Si and C atoms are long enough.

At high total pressures, consequently, SiC films with better crystallinity are grown because the supply rate of Si atoms which are provided only from the dissociation of TMS is low. However, at low pressures, SiC films have worse crystallinity because a large amount of Si atoms are supplied from the cracked TMS and the bulk Si substrate.

In order to prove that a carbonized intermediate layer is necessary for the growth of single crystalline SiC films on Si substrates, SiC layers were grown on carbonized Si substrates. Si substrate was heated up to 1200°C within 1 min with the flows of 1.0 sccm C_3H_8 and 200 sccm H_2, and carbonized for 1 min at 1200°C and 1 atm. The growth of SiC thin film was then carried out over the carbonized layer for 15 min at 1100°C and 275 Torr with the flow rates of TMS and H_2 at 0.5 and 200

sccm, respectively.

Figs. 9(a) and (b) show TEM micrograph and TED pattern of the grown SiC film, respectively, which indicate that a single crystalline SiC(111) film was grown on the carbonized Si substrate without the formations of thick intermediate layer and voids. This result proves that the carbonized intermediate layer is essential to grow a single crystal SiC thin films using TMS, though their size changes depending on deposition pressure.

As the flow rate of TMS increases, grain sizes in SiC layers become smaller and denser, as can be seen in SEM pictures of Fig. 3. When the flow rate of TMS is low, small number of SiC nuclei are formed initially across the Si substrate owing to the low supply of C atoms. The average distance between the nuclei is large and the grown film is less dense, compared to that at high flow rate of TMS. In addition, Si atoms must diffuse out from the bulk of the substrate for the stoichiometric SiC film growth, since the concentration of gaseous Si atoms is only 25% of that of C atoms in the cracked TMS gas. It is believed that this outdiffusion of Si atoms through SiC nuclei during the SiC growth may cause the SiC grains to grow mainly in the direction normal to the surface with sizes of grains approximately unchanged. On the other hand, when the flow rate of TMS is high, nuclei of SiC grains are formed uniformly over the entire surface of the substrate. Therefore the grown SiC film is denser compared to that at the low flow rate of TMS.

Voids are formed in the place where Si atoms diffuse out to the substrate surface, near the SiC/Si interface regardless of the gas pressure. They are similar in shape as those formed during the carbonization of Si substrates using hydrocarbons such as CH_4 and C_2H_2 [Mogab and Leamy, 1974]. The typical size of these voids decreases as the gas pressure increases, as can be seen in Fig. 5. This is believed due to the reduction of the vapor pressure of silicon at higher gas pressures, and hence the reduction of the amount of Si atoms outdiffusing from substrate.

CONCLUSIONS

RTCVD reactor is used to grow epitaxial SiC thin films on Si substrates without carbonization at various substrate temperatures (1000-1100°C), H_2 flow rates (20-200 sccm), and TMS flow rates (0.1-5 sccm). The grown SiC films show the orientation of (111) plane of β-SiC, and free Si and C are not observed in the films. The QMS analysis indicates that TMS is almost completely decomposed at the SiC growth temperature into gaseous Si atoms and CH_3 radicals. Methyl radicals are united to form C_2H_6 molecules, which dissociate rapidly at 1100°C to form mainly CH_4 and C_2H_2 molecules. The formation of voids are observed in the Si side of the SiC/Si interface and the size of the voids decreases with increasing total pressure. A polycrystal SiC films is grown on Si substrates at low pressure. However, at high pressure, an intermediate SiC layer of 18 nm thick is first grown on the Si substrates, and then a single-crystal SiC thin film grows on the layer. Long migration times of absorbed Si atoms and C-containing species are necessary to grow a single crystal β-SiC(111) on Si substrates.

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