2H-SiC Films Grown by Laser Chemical Vapor Deposition

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Abstract We demonstrated the synthesis of 2H-SiC films on graphite and (0001) sapphire substrates by laser chemical vapor deposition. A tris(dimethylamino)silane was used as a novel precursor for the synthesis of SiC films in a CH₄ atmosphere. The 2H-SiC films were obtained at a deposition temperature of 920 K on the sapphire substrate. The films comprised *a*-axis-oriented columnar grains and their in-plane orientation relationship was [1000] 2H-SiC // [0001] sapphire and [0001] 2H-SiC // [1000] sapphire. The films were deposited at the rate of 182 μm h⁻¹.

Keywords: Silicon Carbide; 2H-SiC; Film; Chemical Vapor Deposition

Introduction

Silicon carbide (SiC) is widely used in high-temperature structural materials, protective coatings, and semiconductor devices [1]. SiC has more than 200 polytypes, including zinc blend-type cubic (3C) and many hexagonal structures such as 2H, 4H, 6H, and 15R because of a wide variety of stacking sequence of the Si–C bilayer. Plates of 3C-SiC and 4H/6H single crystalline wafers are commercially produced by chemical vapor deposition (CVD) and sublimation, respectively. However, 2H-SiC films to the best of our knowledge have never been prepared by CVD.

The band gap of 2H-SiC is wide (3.33 eV), as indicated by the absorption and luminescence measurements of 2H-SiC whiskers [2]. This value is larger than that exhibited by 3C-SiC (2.2 eV), 4H-SiC (3.26 eV), and 6H-SiC (3.00 eV) [3]. In addition, Monte Carlo simulations predict a higher electron mobility for 2H-SiC ($1200 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$) than 3C-SiC ($900 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$), 6H-SiC ($370 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$), and 4H-SiC ($720 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$) [3,4]. 2H-SiC is a promising semiconductor material compatible with wurtzite-type group-III semiconductors, such as AlN, GaN, and ZnO.

Only whiskers of 2H-SiC have been obtained by CVD with the pyrolytic decomposition of Si chloride precursors, such as CH_3SiCl_3 or SiCl_4 with $\text{C}_6\text{H}_5\text{CH}_3$ or CCl_4 in a H_2 atmosphere at 1573–1773 K [5–9]. Films of 3C-SiC are mostly obtained by CVD at a wide range of deposition temperatures (1373–2673 K), except for epitaxial growth of 4H- and 6H-SiC films on single-crystal substrates [1,10,11]. Although 2H-SiC may be stable at less than 1673 K [12], 2H-SiC films have never been

synthesized by CVD. Since CVD is a versatile technique to form a conformal coating in a wide area, a novel CVD technique to produce 2H-SiC films should be developed.

Crystalline SiC films can be prepared at a lower deposition temperature by metalorganic precursors than by halide precursors [1]. Although the use of low deposition temperatures often resulted in an amorphous film or absence of deposition [1,13], we have reported that laser irradiation during CVD enables the formation of 3C-SiC films at lower temperatures using an alkylsilane precursor ($\text{Si}_2(\text{CH}_3)_6$) [14,15]. An aminosilane precursor has been used for preparing SiO_2 thin films [16] and amorphous silicon carbonitride films [17]; however, it has never been used for the growth of crystalline CVD-SiC films.

In this study, we demonstrate the preparation of 2H-SiC films on graphite and (0001) sapphire substrates by laser CVD using an aminosilane precursor in a CH_4 atmosphere. The effect of deposition conditions on the orientation and microstructure of the 2H-SiC films were investigated.

Experimental procedure

The CVD apparatus used in this study has been described elsewhere [18]. Tris(dimethylamino)silane (3DMAS, $\text{SiH}(\text{N}(\text{CH}_3)_2)_3$) (Kojundo Chemical, Sakado, Japan) was evaporated at 273 K and the vapors were transported to the deposition chamber using Ar as the carrier gas. CH_4 was introduced into the chamber separately through a double-tube nozzle. The total pressure of the chamber was maintained at 800 Pa. A graphite disc (10 mm in diameter and 1 mm in thickness) and

(0001) sapphire plate (10 mm in size and 1 mm in thickness) were used as substrates. The entire substrate was irradiated with a continuous-wave Nd:YAG laser ($\lambda = 1064$ nm). The deposition temperature (T_{dep}) was measured using a thermocouple. The deposition rate was calculated using the film thickness and deposition time (0.6 ks).

The phase compositions were determined by X-ray diffraction (XRD) (Ultima-IV; Rigaku, Tokyo, Japan) using Cu K α radiation and a silicon strip detector in the range of $2\theta = 20^\circ$ – 80° at the scan speed of $40^\circ \text{ min}^{-1}$. A slow scan was performed with a scintillation counter and a graphite monochromator in the range of $2\theta = 40^\circ$ – 55° at the scan speed of $0.1^\circ \text{ min}^{-1}$. The microstructures were observed with a scanning electron microscope (SEM) (S-3100H; Hitachi, Tokyo, Japan) and a transmission electron microscope (TEM) (EM-002B; Topcon, Tokyo, Japan). The intensity profile by azimuthal integration of selected-area electron diffraction (SAED) pattern was drawn using the Diffraction Ring Profiler software [19]. The schematic of the film crystal structure was created using the VESTA software [20]. The binding energies of the C, Si, and N atoms were measured with X-ray photoelectron spectroscopy (XPS), which was performed using Al K α radiation (Shimadzu-Kratos AXIS-ULTRA DLD). Ar $^+$ ion bombardment was conducted for 1.8 ks. The spectra were fitted with a Gaussian function with baseline correction. The Si–C peak at 100.6 eV was used as the charge reference.

Results and discussion

On graphite substrates, 2H-SiC films were prepared at a deposition temperature of 1010 K (Fig. 1(a)). The 3C-SiC film formed at higher deposition temperatures (> 1057 K). On (0001) sapphire substrate, the 2H-SiC films were prepared at a deposition temperature of 920 K (Fig. 1(b)). Film deposition was absent at temperatures less than 910 K, whereas (111)-oriented 3C-SiC films were obtained at deposition temperatures > 960 K. Despite the overlapping of diffraction peaks of SiC polytypes, the existence of 2H-SiC 011 $\bar{2}$ diffraction ($2\theta = 49.7^\circ$) and the lack of 6H-SiC 011 $\bar{4}$, 011 $\bar{5}$ and 011 $\bar{7}$ diffractions ($2\theta = 41.5^\circ$, 45.3° , and 54.7° , respectively) imply the formation of 2H-SiC.

X-ray pole figures of sapphire 011 $\bar{2}$ and 2H-SiC 101 $\bar{1}$ diffractions of the 2H-SiC film showed threefold patterns at an elevated angle (α) of 57° and 40° (Fig. 2), which corresponded to the interior angle between the (011 $\bar{2}$) and (0001) sapphire planes, and the (101 $\bar{1}$) and (211 $\bar{0}$) 2H-SiC planes, respectively. The X-ray pole figure suggested that the film comprised (211 $\bar{0}$)-oriented, namely *a*-axis-oriented, 2H-SiC grains with an in-plane orientation relationship of [1000] 2H-SiC // [0001] sapphire and [0001] 2H-SiC // [1000] sapphire (Fig. 2(b)).

The 2H-SiC film showed a columnar structure on the (0001) sapphire substrate and the deposition rate of the film was $182 \mu\text{m h}^{-1}$ (Fig. 3(a)). The surface microstructure of the 2H-SiC film was characterized by fine grains with a star flake-like structure (inset, Fig. 3(a)). A bright-field TEM image showed that an equiaxed grain structure was formed on the substrate, and columnar grains developed at later stages of deposition (Fig. 3(b)). In CVD, rapid deposition often results in the preferred growth of

columnar grains, which subsequently develop into equiaxed grain structures on the substrate [21]. An SAED pattern taken from the equiaxed grain structure showed a Debye–Scherrer ring (Fig. 3(c)). The ring pattern and corresponding intensity profile (inset, Fig. 3(c)) were indexed as 2H-SiC. The existence of 2H-SiC 0112 diffraction ($d^{-1} = 0.55 \text{ nm}^{-1}$) supports the formation of 2H-SiC structure, as well as the XRD results. The SAED pattern of the columnar grain belonged to the $[11\bar{2}1]$ zone axis, confirming an a -axis-oriented growth of the 2H-SiC columnar grains (inset, Fig. 3(b)). A high-resolution TEM image of the columnar grain taken along the $[0001]$ zone axis showed a hexagonal lattice of the (0001) 2H-SiC plane (Fig. 3(d)). No stacking faults were observed in the 2H-SiC columnar grains.

The CVD synthesis of 2H-SiC whiskers on graphite susceptor has been reported with a narrow synthesis temperature window (30–100 K) at 1573–1773 K [5–9]. Several studies claim that 2H-SiC whiskers grow via the vapor-liquid-solid (VLS) mechanism similar to Si whiskers because of the presence of spherical terminations and joints [8,9]. Later studies have reported that spherical precipitation prevents the whisker growth, rather than associating with VLS growth [2]. TEM analysis in the present study shows no evidence for VLS growth.

Figure 4 shows the C 1s, Si 2p, and N 1s XPS spectra for the 2H-SiC films prepared on the (0001) sapphire substrate. Each XPS spectrum was deconvoluted into two Gaussian peaks, namely, peaks at 283.4 and 284.5 eV in the C 1s spectrum (Fig. 4(a)), peaks at 100.6 and 101.7 eV in the Si 2p spectrum (Fig. 4(b)), and peaks at 397.6 and 398.7 eV in the N 1s spectrum (Fig. 4(c)). The peak assigned to O–Si

(532 eV) in the O 1s spectrum was background level (inset, Fig. 4(c)).

The peaks at 100.6 and 283.4 eV corresponded to the bonding states of Si–C₄ (100.6 eV [22]) and C–Si (283.4 eV [22]) in SiC, respectively. The peak at 284.5 eV was associated with the C–C bonding between C-rich SiC [23] and a free carbon. The peaks at 101.7 and 397.6 eV were consistent with the Si–N₄ (101.7–101.9 eV [24,25]) and N–Si (397.8 eV [24,25]) bonding states in Si₃N₄, respectively. A small shoulder at 398.7 eV in the N 1s spectrum might be related to the incorporation of an NH species (Si–NH–Si) or an NH₂ fragment bonded to Si (Si–N=(H)₂) [26] rather than a N atom bonded to C, owing to the absence of a C–N peak at 286.5 eV [27] in the C 1s spectrum.

The Si 2p peak was fitted with Si–C₄ and Si–N₄ peaks that have narrow full-width and half-maxima (1.6 eV and 1.3 eV) that are comparable to the reference values (1.8 eV [22] and 2.1 eV [25]). Although C atoms in SiC might be substituted by N, no intermediate bonding states of Si–C–N were identified. N concentration in SiC is commonly on the order of 10¹⁵–10²⁰ cm^{–3}, which is much lower than the detection limit of XPS [22]. On the other hand, a N concentration close to the solubility limit (10¹⁹–10²⁰ cm^{–3}) resulted in the formation of stacking faults and a phase transformation from 4H to 3C [30–32]. First principles calculations also implied that substitution of C with N atoms destabilized 6H-, 4H-, and especially 2H-SiC [33]. Therefore, N concentration in SiC might be less than the solubility limit at least. Since the XRD and TEM-SAED patterns revealed no evidence for crystalline Si₃N₄, most of the nitrogen likely coexisted in the 2H-SiC films as a trace amount (3 mol%) of amorphous Si₃N₄ rather than

N-substitution in SiC.

According to the thermal stability diagrams of the SiC polytypes summarized by Knippenberg [12] and Inomata *et al.* [34], 2H-SiC may be stable at less than 1673 K [12]. However, 2H-SiC were only obtained as whiskers, which grew under a large temperature gradient. 3C-SiC films have mostly been obtained by CVD over a wide temperature range (1373–2673 K). This might be attributed to the symmetric tetrahedral and octahedral shapes of 3C-SiC nuclei having lower surface energies than the hexagonal prismatic nuclei of 4H- and 6H-SiC polymorphs while in the gas phase [34,35] and in particular a highly supersaturated Si-excess atmosphere [34]. Conversely, first principles calculations suggested that a Si vacancy may stabilize 2H-SiC [33]. Therefore, a Si-poor environment and a relatively large temperature gradient established by laser irradiation would favor the formation of 2H-SiC films. It is noteworthy that while 2H-SiC films have never been prepared by conventional CVD, we report the possibility of preparing 2H-SiC films by laser CVD using 3DMAS precursor and CH₄ atmosphere.

Conclusions

By using laser CVD with 3DMAS precursor, films of 2H-SiC were prepared on graphite and (0001) sapphire substrates at deposition temperatures of 1010 K and 920 K, respectively. The typical deposition rate was 182 $\mu\text{m h}^{-1}$. The 2H-SiC films prepared on (0001) sapphire substrates were composed of *a*-axis-oriented 2H-SiC columnar grains. The in-plane orientation relationship of the *a*-axis-oriented

2H-SiC grains was [1000] 2H-SiC // [0001] sapphire and [0001] 2H-SiC // [1000] sapphire. A trace amount of amorphous Si₃N₄ might coexist in the 2H-SiC films.

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Figure captions

Figure 1 XRD patterns of the 2H-SiC films prepared (a) on graphite substrates at 1010 K, and (b) on (0001) sapphire at 920 K. Inset patterns were measured at a slow scan condition. Reference lines: 2H-SiC (ICSD No. 86253), 3C-SiC (No. 24271), and 6H-SiC (ICSD No. 156190).

Figure 2 (a) X-ray pole figures of $(01\bar{1}2)$ sapphire and $(10\bar{1}1)$ 2H-SiC planes of the 2H-SiC film. (b) Schematic of the in-plane orientation relationship between $(2\bar{1}\bar{1}0)$ 2H-SiC and (0001) sapphire planes.

Figure 3 (a) Cross-sectional and (inset in a) surface SEM images of the 2H-SiC film prepared on (0001) sapphire substrate. (b) Bright-field TEM image and (inset in b) SAED pattern taken from columnar grain along the $[11\bar{2}1]$ zone axis. (c) SAED pattern taken from equiaxed grain structure and reference line of 2H-SiC structure. Inset in (c) shows the intensity profile of the SAED pattern. (d) High-resolution TEM images of the 2H-SiC columnar grain taken along the $[0001]$ zone axis and corresponding atomic arrangement.

Figure 4 (a) C 1s, (b) Si 2p, and (c) N 1s XPS spectra for the 2H-SiC film prepared on a (0001) sapphire substrate. The inset in c shows the O 1s XPS spectrum.

Fig.01 XRD patterns of 2H-SiC films

Fig.02 X-ray pole figure of 2H-SiC films

Fig.03 SEM and TEM images of 2H-SiC films

Fig.04 XPS spectra of 2H-SiC films

Figure captions

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