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Chemical Vapor Deposition of Single Crystalline β-SiC Films on Silicon Substrate with Sputtered SiC Intermediate Layer

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ABSTRACT

A single crystal of β-SiC was grown by chemical vapor deposition using an SiH_4-C_3H_8-H_2 system on a silicon substrate with a sputtered layer. The grown layer of 4 μm thickness was confirmed as a single crystal by examination with reflection electron diffraction and x-ray diffraction. To reduce the large mismatch between β-SiC and a silicon substrate, a sputtered β-SiC layer was employed as a buffer layer. Even though the sputtered layer was polycrystalline, the subsequent layer deposited by CVD was a single crystal. The crystallinity of the deposited layer was strongly affected by the thickness of the sputtered layer, the substrate temperature during sputtering, and the temperature of chemical vapor deposition.

Silicon carbide (SiC) is an interesting material for electronic and optical device applications because of its high temperature stability and large energy gap. The cubic form, β-SiC, has a bandgap energy of 2.35 eV (1) at room temperature, an electron mobility of 1000 $cm^2/V \cdot sec$ (2), and a drift velocity of 2.7×10^7 cm/sec (at 2×10^5 V/cm) (3). These properties are particularly attractive for minority carrier as well as majority carrier device applications. However, a large area substrate is not available because of the difficulty of crystal growth. Crystals of β-SiC obtained by a sublimation process (Lely method) have small size and irregular shapes, thus complicating wafer processing for device fabrication. In order to obtain β-SiC single crystals of large area, it is desirable to grow β-SiC on foreign substrates.

Epitaxial growth of β-SiC on foreign substrates has been reported by many investigators with chemical vapor deposition (CVD) (4-6), chemical conversion (7-9), evaporation (10), or rf sputtering methods (11-13). They, however, reported only very thin films of β-SiC. By the chemical conversion method. Nakashima *et al.* (9) have obtained single crystal films of β-SiC less than 100 nm thick on an Si substrate. Examination with reflection electron diffraction (RED) showed the change of the crystal structure from single crystal to polycrystal with increasing film thickness. Rohan *et al.* (5) showed oriented β-SiC layers grown on sapphire substrates by CVD and reported that the growth of single crystal films was not possible because of relatively large lattice mismatch.

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Key words: silicon carbide, CVD, epitaxial films, thin films, sputtering.

Difficulty with heteroepitaxial growth comes from the lattice mismatch between the grown layer and the substrate (a_{SiC}: 4.358Å, a_{Si}: 5.430Å, $a_{sapphire}$: 4.75Å, $c_{sapphire}$: 12.95Å). One of the approaches to overcome this difficulty is to introduce a buffer layer between substrate and deposit. This approach is well known in the crystal growth of III-V compounds by liquid phase epitaxy. A few examples are reported for CVD systems such as GaP/evaporated P/Si (14), ZnO/sputtered ZnO/sapphire (15), β-SiC/evaporated Ni/α-SiC (16), and α-SiC/sputtered AlN/W (17).

We previously reported the preparation of β-SiC on Si substrates by a sputtering method and suggested that the sputtered β-SiC layer might be attractive as a substrate in the CVD process (18). In the CVD process, we reported the optimum conditions for epitaxial growth of β-SiC on Si substrates with an $SiCl_4$-C_3H_8-H_2 system (19). In this report, chemical vapor deposition of single crystal β-SiC films on Si substrates with sputtered β-SiC intermediate layers is described. Chemical vapor deposition was carried out using an SiH_4-C_3H_8-H_2 system. The crystallinity of the deposited layer was found to be strongly affected by the thickness and the crystalline structure of the sputtered layer. The surface morphology and crystallinity of the layer grown by CVD are described. The growth mechanism of β-SiC is also discussed.

Experimental

Sputtering.—Thin films of β-SiC were deposited on Si substrates by rf sputtering. The target material was an SiC disk from Union Carbide Company. The distance between the target and the substrate was 4.5 cm. The Si substrate was heated by a molybdenum strip heater and the temperature was measured by means of an optical pyrometer.

After preliminary evacuation of the reaction chamber to 1×10^{-6} Torr, argon gas was introduced up to a pressure of 1×10^{-2} Torr. Rf power of 200W (peak voltage of 2 kV and frequency of 13.5 MHz) was applied. Substrate temperatures were varied between 800° and 1000°C. Sputtering time was varied from 100 sec to 60 min. The thickness of the thin film was determined by using an interference microscope at a step made by masking a portion of the substrates during sputtering.

Chemical vapor deposition.—The deposition of β-SiC by pyrolysis of silane (SiH_4) and thermal decomposition of propane (C_3H_8) in a hydrogen flow system was carried out using the apparatus shown in Fig. 1. The reaction tube of 30 mm ID and 440 mm length was made of clear-fused quartz with a water-cooled jacket. The thickness of the sputtered layer was 30-80 nm. After being rinsed in acetone and dried, the substrate was put on an SiC-coated graphite susceptor ($24 \times 5 \times 30$ mm^3). To obtain a uniform grown layer, the susceptor was tilted at an angle of 10° from the horizontal. The linear velocity (defined as the ratio of the carrier gas flow rate to the cross section of the reaction tube) of the system was approximately 2 cm/sec. The temperature was measured by observing the Si substrate through the water-cooled wall of the reaction tube with an optical pyrometer. The measured temperature was calibrated taking account of the emissivity of Si, but no correction was made for absorption by the water-cooled quartz jacket. The entire system was evacuated by a rotary pump and backfilled with H_2. After H_2 flowed for 10 min, the substrate was heated externally by an rf generator and kept at 1000°C for 5 min before deposition of β-SiC.

The deposition of the β-SiC was carried out at a substrate temperature in the range of 1000°-1360°C with the standard gas composition: H_2-750 cm^3/min, SiH_4-0.6 cm^3/min, C_3H_8-0.2 cm^3/min, Si/H_2-0.7 $\times 10^{-3}$, and $Si/C = 1$. The deposition time was usually 30-180 min. The thickness of the deposited β-SiC films was determined by direct observation of the cross section of fractured specimens with a scanning electron microscope (SEM). To study the crystallinity of the β-SiC films, the grown layers were examined by reflection electron diffraction with an acceleration voltage of 80 kV. The lattice parameters of the grown layers were determined from a spot pattern using the diffraction pattern of gold as a reference.

Experimental Results

Preparation of substrates.—The relation between the thickness of the sputtered SiC film and sputtering time is shown in Fig. 2. The sputtering rate depends on the substrate temperature. The higher the substrate temperature, the lower the sputtering rate obtained. The temperature dependence is explained by a reduced sticking coefficient of sputtered atoms at high substrate temperatures, or by decreased sputtering yield resulting from high target temperature due to thermal radiation from the heated substrate (20).

Since the substrate with the sputtered layer is kept at high temperature during the CVD process, the crystallinity of the sputtered layer could be changed by the heating process. Consequently, the crystallinity of the sputtered layer before and after annealing was examined by RED analysis. Thermal annealing was carried out in an Ar ambient at 1270°C for 30 min. Diffraction patterns of the sputtered layer before and after annealing are shown in Fig. 3. Before annealing, every RED pattern showed haloes which implies an amorphous state. This was true even if a high substrate temperature was employed during sputtering, as shown in Fig. 3(b) and (c). After annealing, the RED pattern showed Debye rings which imply a polycrystalline state. The Debye rings became stronger

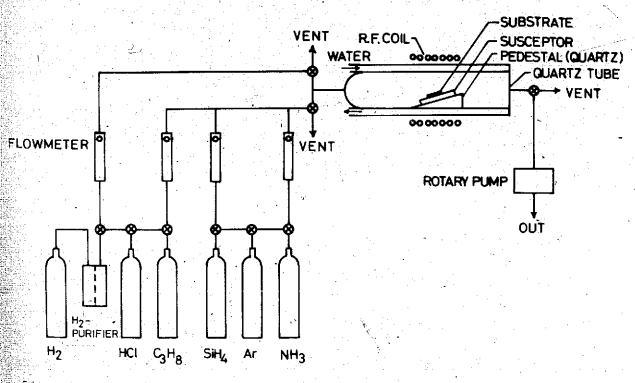

Fig. 1. A schematic diagram of the reaction tube and CVD system.

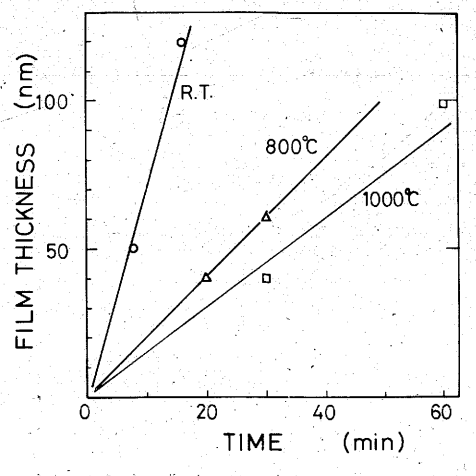

Fig. 2. Relation of film thickness and deposition time for sputtered films prepared at various substrate temperatures: ○, room temperature; △, 800°C; □, 1000°C.

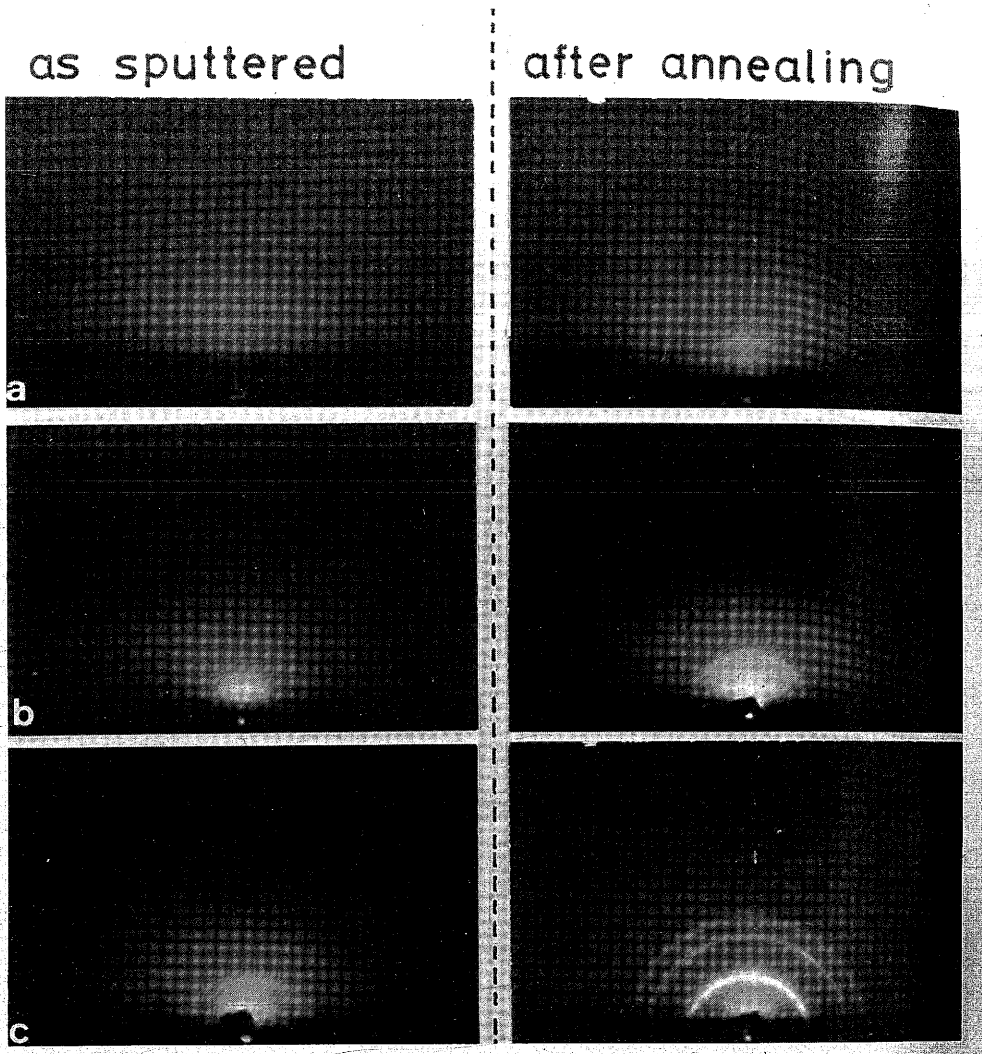

Fig. 3. Reflection electron diffraction patterns from layers as-sputtered and annealed at 1270°C for 30 min in Ar ambient. Substrate temperatures during sputtering were (a) room temperature, (b) 800°C, and (c) 1000°C.

when the substrate was kept at high temperature during sputtering. The sputtered layer was verified as being polycrystalline β-SiC by analyzing the RED pattern.

Temperature dependence of the CVD growth rate.— The growth rate as a function of the substrate temperature is plotted in Fig. 4. Vertical bars in the figure define the thickness difference from run-to-run. The mole ratio of the reactant gas (SiH_4, C_3H_8, Si/C = 1) to hydrogen is shown in the figure. The open circles indicate a higher mole ratio than that for the solid circles. While the higher mole ratio gave higher growth rate, it also gave an irregular surface and poor crystallinity, so the layer grown with the higher mole ratio is not favorable for device applications. The lower mole ratio gave a smooth surface and good crystallinity; therefore the ratio represented by a solid circle in Fig. 4 was chosen as a standard gas composition. The growth rate is expressed by an exponential function of the reciprocal substrate temperature above 1200°C, which indicates that the growth rate is limited by surface reaction. The activation energy for the formation of β-SiC calculated from the slope of the line is about 15 kcal/mole. An activation energy of 20-25 kcal/mole was obtained for the growth of β-SiC on an Si substrate without a sputtered layer when using the $SiCl_4$-C_3H_8-H_2 system (19). Two reasons may be given for the two activation energies. First, the growth of β-SiC on an Si substrate is heteroepitaxial growth, while the growth of β-SiC on a buffer layer (polycrystalline β-SiC) is homoepitaxial growth to some extent. The activation energy for homoepitaxial growth is generally smaller than that for heteroepitaxial growth. Second, $SiCl_4$ was used for the Si source in the previous work (19), while SiH_4 was used in this work. Since the temperature of decomposition of SiH_4 is lower than that of $SiCl_4$, a smaller activation energy might be expected.

The growth rates appear to be constant at substrate temperatures lower than 1200°C. The deposition mech-

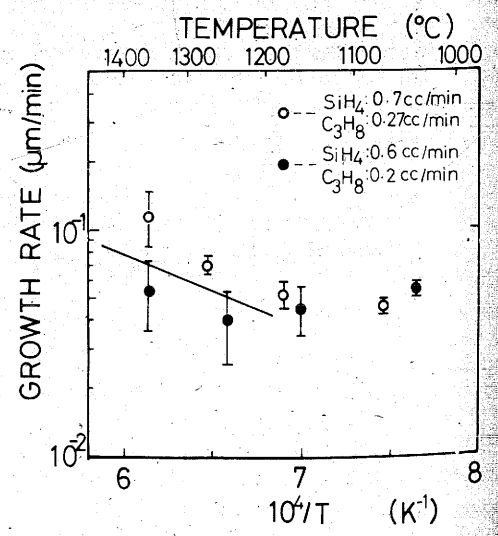

Fig. 4. Temperature dependence of the CVD growth rate

anism might be different in the high substrate temperature and low substrate temperature regions. Although the sputtered layer originates many nucleation sites for subsequent CVD growth, the layers grown by CVD at low temperatures tend to be polycrystalline because of short migration length of adatoms on the surface.

Surface morphology.—Surface morphology of the layers grown at three substrate temperatures is shown in Fig. 5. When the deposition was done at 1070°C, the surface was covered with rounded grains of size less than 0.2 μm. On the surface grown at 1250°C, the size of the rounded grains became larger. When the film was deposited at 1360°C, the surface was strongly influenced by the crystal habit of the substrate. Although the size of the grains became larger at a substrate temperature lower than 1360°C, the layer showed a bumpy surface resulting from incomplete coalescence of the grains. When the substrate temperature was above 1360°C, coalescence of the grains was fully developed and a replica of the crystal habit of the substrate appeared in the grown layer. Figures 6(a) and (b) show surface morphology of the layer grown at 1360°C. Square crystallites with a side of 1 μm were observed on the layer grown on the (100)Si substrate, while triangular crystallites with a side of 2 μm were observed on the layer grown on the (111)Si substrate.

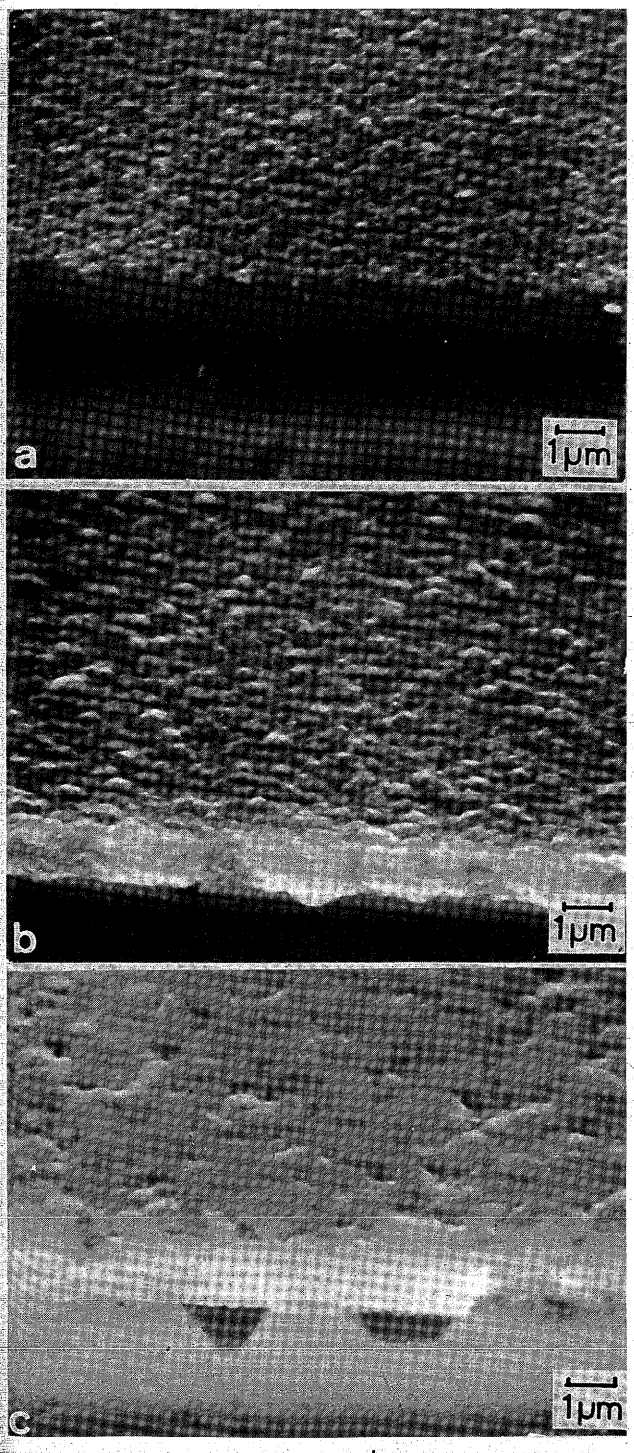

Fig. 5. Morphology of the grown layer for different substrate temperatures: (a) 1070°C, (b) 1250°C, (c) 1360°C.

Crystalline properties of β-SiC films.—The crystallinity of growth obtained by the CVD process was strongly affected by the thickness of the sputtered SiC layer and the substrate temperature during sputtering. The layer grown by CVD was not a single crystal when the sputtered layer was thicker than 100 nm, even if a high substrate temperature was employed during sputtering. When the sputtering was done at a substrate temperature lower than 800°C, the subsequent layer grown by CVD was not a single crystal in spite of keeping the substrate at high temperature during the CVD process. From these experiments, it was found that the Si substrate should have a sputtered β-SiC overlayer with a thickness of 30-80 nm prepared at a substrate temperature of 1000°C.

The growth temperature for CVD is one of the important parameters which determines the crystallinity of the grown layer. The crystallinity of a layer of about 4 μm thickness was examined by the RED method. The diffraction pattern of β-SiC prepared at 1160°C showed so-called Debye rings which indicate a polycrystalline structure. Spots were superimposed

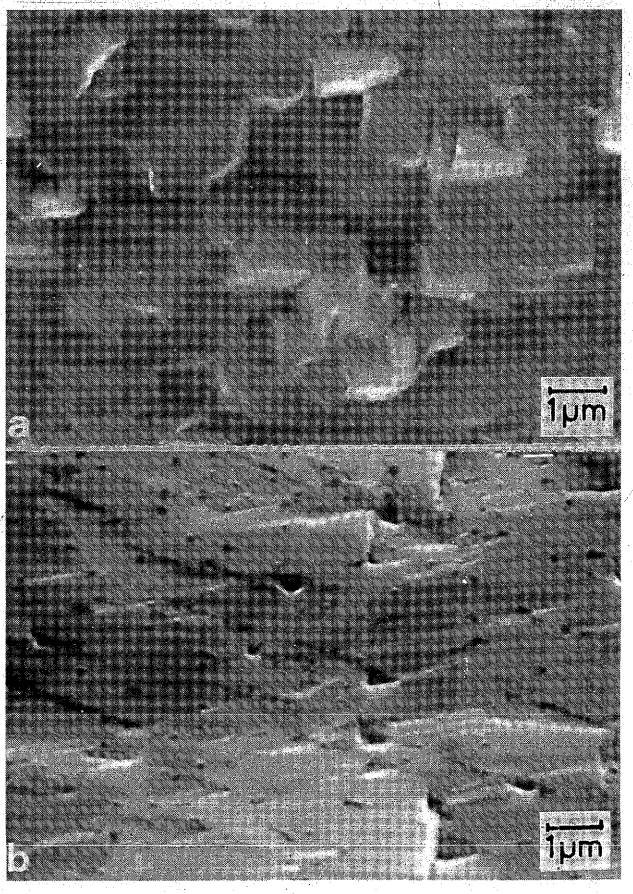

Fig. 6. Morphology of the grown layer reflecting the crystal habit of the substrate. (a) Layer grown on (100)Si substrate with a sputtered layer. (b) Layer grown on (111)Si substrate with a sputtered layer.

on the faint Debye rings for the film prepared at 1250°C, but the diffraction pattern was not changed when the direction of the incident electron beam was changed. The grown layer was therefore considered to have a preferred orientation. For the film prepared at 1360°C, the diffraction pattern consisted of strong spots. Consequently, it may be concluded that a change from polycrystalline to single crystal structure occurred with increasing substrate temperature. Good single crystals seem to be obtained at approximately 1360°C.

To investigate the substrate-orientation dependence of the growth, substrates with (111) or (100) surface planes were used. The CVD growth of β-SiC was carried out on the two substrates simultaneously using the standard gas composition at 1360°C. The diffraction patterns from the layers grown on the two substrates are shown in Fig. 7. Strong spots indicate that the layers are single crystalline. The orientation relationship between the overgrowth and the substrates is as follows

$$\beta\text{-SiC}(111)//\text{Si}(111), \beta\text{-SiC}[\bar{1}10]//\text{Si}[\bar{1}10],$$

$$\beta\text{-SiC}(100)//\text{Si}(100), \beta\text{-SiC}[001]//\text{Si}[001]$$

Since the RED measurement gives information on crystallinity very near the surface, the layer was examined by x-ray diffraction with a copper target for further crystal analysis. Figure 8 shows typical diffraction charts for the β-SiC layers. A strong peak appeared at $2\theta = 41.40°$ for the layer grown on the (100) substrate. A peak for the Si substrate also appeared due to the thin β-SiC layer. A strong peak also appears at $2\theta = 36.65°$ for the layer grown on the (111) substrate. Furthermore, the β-SiC film without a substrate was also examined by x-ray transmission Laue method. The Si substrate was removed by chemical etching using an HNO_3-HF solution. The Laue photographs are shown in Fig. 9 for the layers grown on the (100) and (111) substrates. The Laue pattern for the layer grown on the (100) substrate shows fourfold symmetric spots with faint Debye rings as shown in Fig. 9(a). The Laue pattern for the layer grown on the (111) substrate shows threefold symmetry with diffuse spots and faint Debye rings, as in Fig. 9(b).

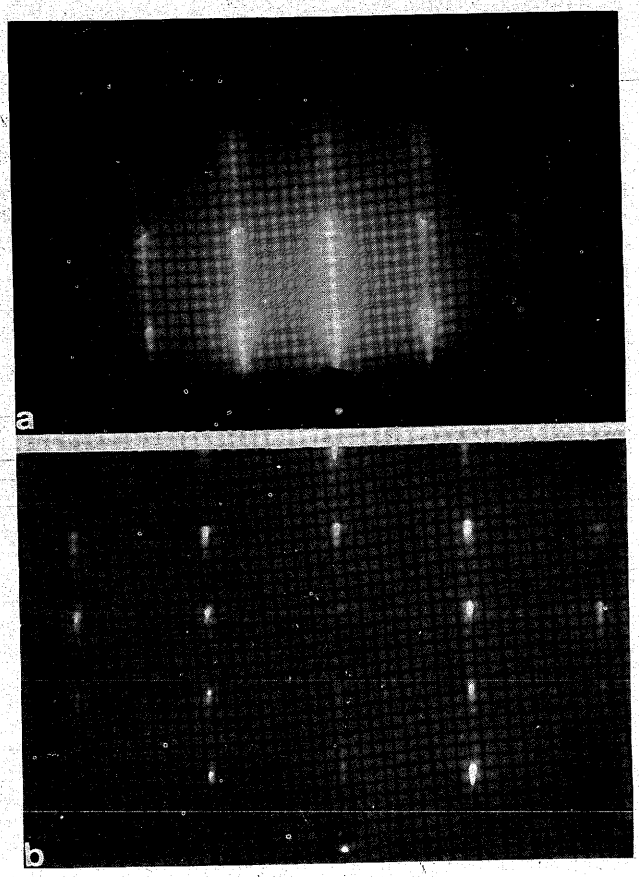

Fig. 7. Reflection electron diffraction patterns from layers grown on (a) (100)Si and (b) (111)Si substrates with sputtered layers.

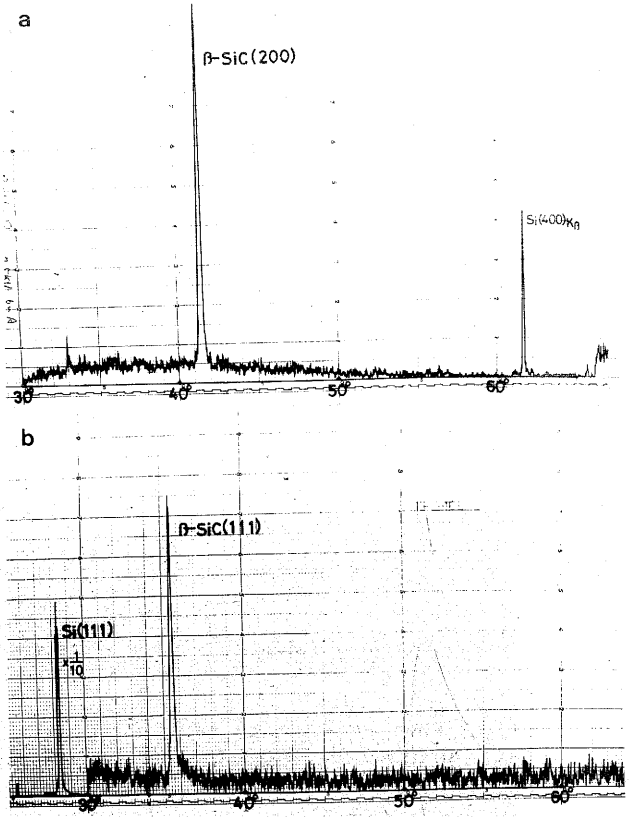

Fig. 8. X-ray diffraction spectrum obtained from layer grown on (a) (100)Si and (b) (111)Si substrates with sputtered layers.

The faint Debye rings are found to arise from the polycrystalline layer contiguous to the Si substrate. The underside of the β-SiC film was observed by SEM and examined by RED. A triangular morphology was observed on the underside surface of the layer grown on the (111) substrate, as shown in Fig. 10(a). In this figure, the length of the side of the triangles was scattered from 0.1 to 0.4 μm. The RED pattern from the underside of the layer shows Debye rings of polycrystalline β-SiC, as shown in Fig. 10(b).

Discussion

When a β-SiC film is deposited directly on an Si substrate, it is difficult to grow single crystals of β-SiC thicker than 100 nm, as reported by most researchers. The difficulty arises from the large lattice mismatch and stresses between the grown layer and the substrate. In this work, by introducing an intermediate layer as a buffer layer, stresses caused by the lattice mismatch could be released. The buffer layer should have an appropriate film thickness and crystallinity, because the crystal field of the substrate must be transferred through the buffer layer.

We speculate that the nature of the polycrystalline β-SiC layer obtained by sputtering is different from the one obtained by CVD. In the CVD system, the grown layer is formed under a condition of thermodynamic equilibrium, so that nucleation and growth are strongly dependent on the surface treatment of the substrate. The initial stage of growth is influenced by parameters difficult to control, such as surface roughness and dislocations. An adatom moves toward favorable sites of the substrate, so the density of nu-

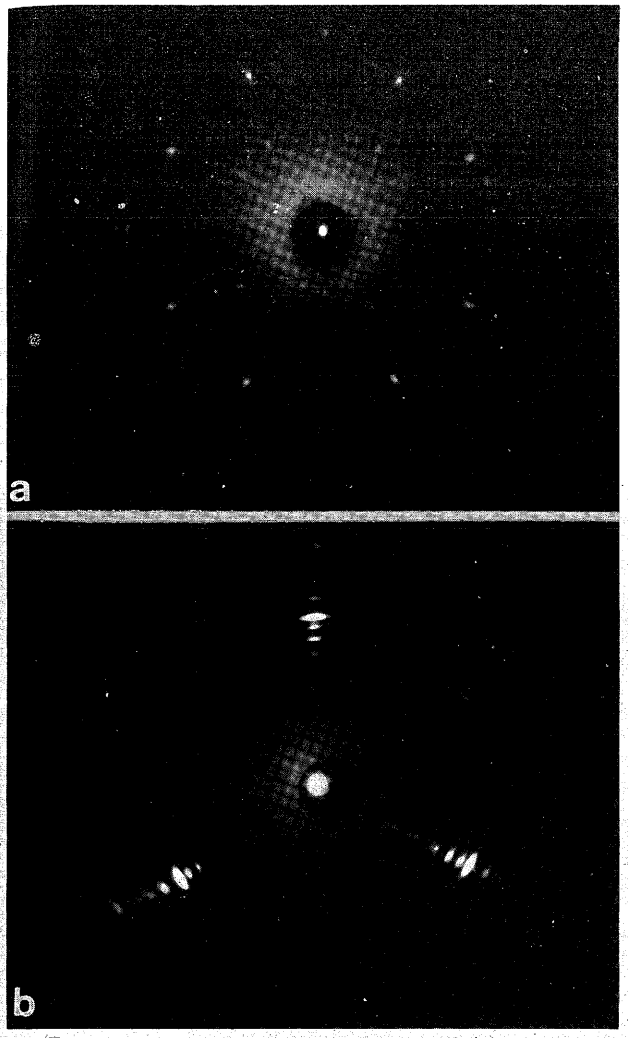

Fig. 9. X-ray transmission Laue photographs of the grown layer removed from (a) (100)Si and (b) (111)Si substrates with sputtered layers.

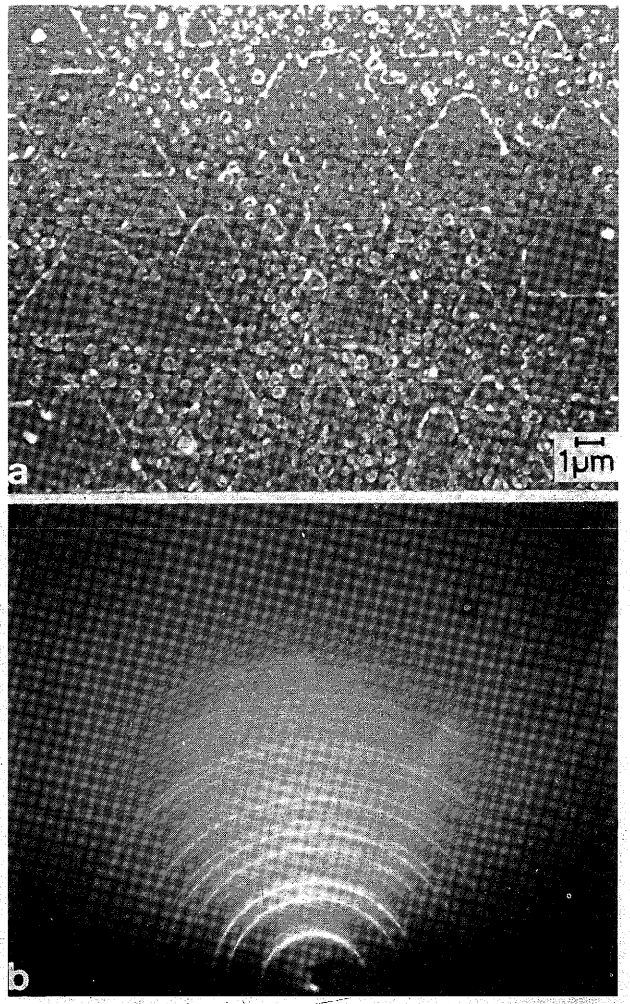

Fig. 10. Crystal structure of the underside of β-SiC after removal of the Si substrate. (a) SEM photograph and (b) RED pattern.

cleation on the surface might be nonuniform atomically. As a result, single crystal growth of β-SiC on an Si substrate by the CVD process needed careful preparation including SiC coating of the susceptor and etching of the substrate before the crystal growth. Furthermore, reproducibility of single crystal growth was not good. In the sputtering method, deposition is carried out under a nonequilibrium condition. Sputtered atoms uniformly hit the whole area of the substrate and nucleation may occur more uniformly over the surface. Therefore, the intermediate layer of β-SiC prepared by sputtering can be more uniform than that by CVD. Consequently, the initial stage of growth in the subsequent CVD process is not strongly dependent on the surface and single crystals of β-SiC are reproducibly obtained. When the CVD deposition was done at a substrate temperature higher than 1360°C, single crystalline β-SiC was also obtained in cases where the film thickness was less than 4 μm.

Conclusion

Single crystals of β-SiC were obtained reproducibly by CVD at a substrate temperature of 1360°C on Si substrates by using sputtered SiC layers as buffer layers. Crystallinity of the β-SiC layer so grown was examined by the RED and x-ray diffraction transmission Laue methods. The epitaxial relation between the β-SiC film and the Si substrate was obtained. Sputtering should be done at a substrate temperature between 800° and 1000°C. To obtain a single crystal by CVD, the thickness of the sputtered layer must be less than 100 nm. It is speculated that the growth of the single crystalline β-SiC was controlled by the crystal field of the Si substrate through a polycrystalline intermediate layer formed by sputtering.

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Application of the Langmuir Probe in Sputtering Techniques

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ABSTRACT

During sputtering the plasma potential of the glow discharge may be influenced by unprotected electrodes situated in the vicinity of the plasma region. A positive electrode potential (V_a) is known to shift the plasma potential by a value of V_a in the positive potential direction. A negative electrode potential has no such influence on the plasma potential. A consequence of this behavior of the plasma potential is that all grounded parts of the vacuum system will establish a substantial ($\sim V_a$) negative potential with respect to the plasma if a positive electrode is introduced. This effect may cause excess contamination of the sputtered films since sputtering during this condition takes place both from the grounded vessel surfaces and from the sputtering target. Also, grounded substrates will then be subjected to unintended bombardment by energetic ions. This is especially important to consider in planar magnetron sputtering, where no energetic bombardment of substrates is supposed to take place. Introducing a negatively biased Langmuir probe into the plasma makes the probe current very sensitive to plasma conductance without affecting plasma potential conditions during sputtering. It is thus possible to monitor the sputter-cleaning of targets. During cleaning of an Al target the secondary emission factor of electrons caused by ion impact changes considerably. Thus the conductance of the plasma changes. This is detected by the Langmuir probe.

Numerous papers have been published in the field of Langmuir probe studies of glow discharges (1-3). We will restrict ourselves to the region of interest during sputtering. It has been reported that during sputtering of Ta and TaO it is necessary to reproduce not only pressure and bias conditions but also plasma conditions in order to obtain reproducible results.

This points out the importance of knowing and understanding plasma conditions during sputtering. It is well known that the plasma potential follows the potential of a positive electrode introduced into the plasma. In this way Holland (4) used the effect for treating and passivating vacuum systems. Coburn and Kay (5) measured the energy of ions extracted from a plasma and showed that the ion energy increased by the same value as a positive bias applied to an electrode exposed to the plasma. Both of these observations strongly confirm that the plasma *I-V* characteristic is indeed affected by an extra electrode introduced into the plasma in a sputtering system.

In some plasma applications it is very important to avoid any possible sputter bombardment of the substrates. If such bombardment takes place it may ruin the performance of the device. We are going to point out some possible sources causing such (unintended) bombardment of the substrates during sputtering.

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Key words: sputtering, discharge, plasma potential.

Experimental Results and Discussion

A system such as that shown in Fig. 1 was used during the probe *I-V* measurements. A single Langmuir planar probe was inserted into the sputtering plant. The upper electrode in Fig. 1 formed the cathode and consequently operated as the target during sputtering. A metal screen was inserted inside the glass bell jar. This metal screen was insulated from ground and a positive or negative voltage may be applied.

Consider the experimental *I-V* characteristics shown in Fig. 2. Here the Langmuir probe characteristics have been measured under different conditions wherein voltage is applied to the metal screen inside the vacuum chamber during sputtering. The *I-V* characteristic clearly shifts to more positive potentials with a value equal to that applied to the metal screen. Since the plasma is quite conductive, the plasma potential V_p follows the potential of the positive metal screen as expected. The implication of this effect is clearly demonstrated by applying a very high positive value to the metal screen. If so, a "dark space" region is observed over grounded areas inside the vacuum cham-