

## Surfaces

### **Atomic Scale Passivation of SiC Surfaces (Invited)**

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### **Oxidation States Present on SiC(0001) after Oxygen Exposures**

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### **A High Resolution Photoemission Study of Hydrogen Terminated 6H-SiC Surfaces**

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### **In-situ Analysis of Thermal Oxidation on H-terminated 4H-SiC Surfaces**

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### **Selective Epitaxial Growth of Pyramidal 3C-SiC on (Late News) Patterned Si Substrate**

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## Atomic Scale Passivation of SiC Surfaces

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Surface passivation is a central issue in successful SiC device applications. It could be achieved by oxidation but also by hydrogenation. In this presentation, I will discuss some important points of latest studies in SiC passivation such as the i) role of atomic defects, ii) oxygen/hydrogen atomic adsorption sites, iii) propagation of the oxidation reaction and H or O surface migration, iv) abrupt insulator/SiC interface formation and new Si phase on hexagonal SiC and v) role of the polytype and of surface reconstruction, composition and temperature.

The initial oxidation of hexagonal (6H,4H) SiC surfaces is investigated by combining three different techniques: core level photoemission spectroscopy (CLPS) using 3rd generation synchrotron radiation source, atom-resolved scanning tunneling microscopy (STM) and infrared absorption spectroscopy (IRAS using  $^{16}\text{O}$  and  $^{18}\text{O}$ ) [1]. High quality clean Si-rich 6H/4H-SiC(0001) 3x3 surfaces are prepared having a very low atomic defect density (better than 2%). In strong contrast to the cases of silicon and 3C-SiC surfaces [2], initial defects (bright and dark) are not adsorption sites for oxygen atoms but result from electronic effects. Oxygen atoms interaction takes place away from the trimer-ad atom (Tri-Ad) well below the surface with oxide product formation already at extremely low oxygen exposures and with oxygen atoms in bridge bonded positions [1]. Also, again in strong contrast to Si, this initial oxidation is likely to relax the highly strained SiC surface [1]. Abrupt SiO<sub>2</sub>/SiC interfaces could be obtained at 500°C when the oxidation is performed on a pre-deposited Si overlayer on the SiC surface [3]. Most interestingly, this Si overlayer is found to have a novel and unexpected cubic 4x3 array growing on a hexagonal SiC surface and to be highly sensitive to oxygen [4].

Molecular H<sub>2</sub> interaction with 3C-SiC(100) 3x2 and c(4x2) surfaces and with Si atomic lines is investigated by atom-resolved STM and valence band photoemission [5]. While the 3x2 surface reconstruction remains totally inert, the c(4x2) is highly reactive to H<sub>2</sub> with sticking probabilities up to 8 orders of magnitude higher than for Si(100)2x1. H<sub>2</sub> is initially dissociated at up-dimer adsorption sites influencing the two neighbor down-dimers. At higher exposures, H induces a 2x1 surface transformation. Interestingly, investigating H atom surface migration, we observe a strong anisotropy with H atom hopping along the dimer rows of the c(4x2) surface (AUDD) and not perpendicularly to the dimer rows. The very high reactivity difference between 3x2 and c(4x2) allows non-reacted Si atomic lines formation on a hydrogenated surface [5].

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**OXIDATION STATES PRESENT ON SiC (0001) AFTER OXYGEN EXPOSURES**

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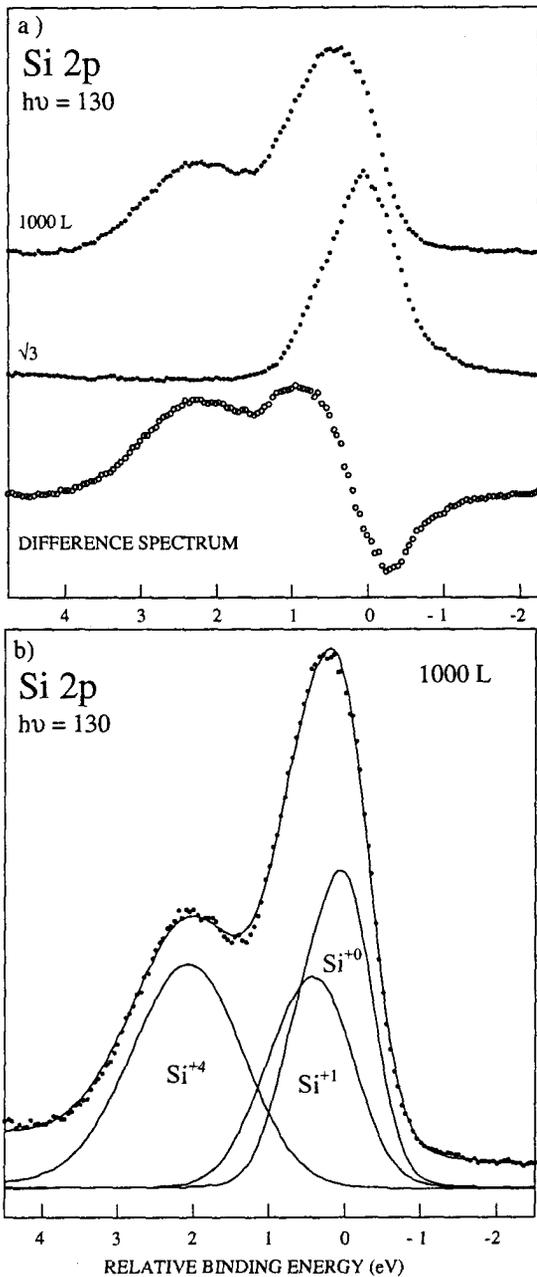
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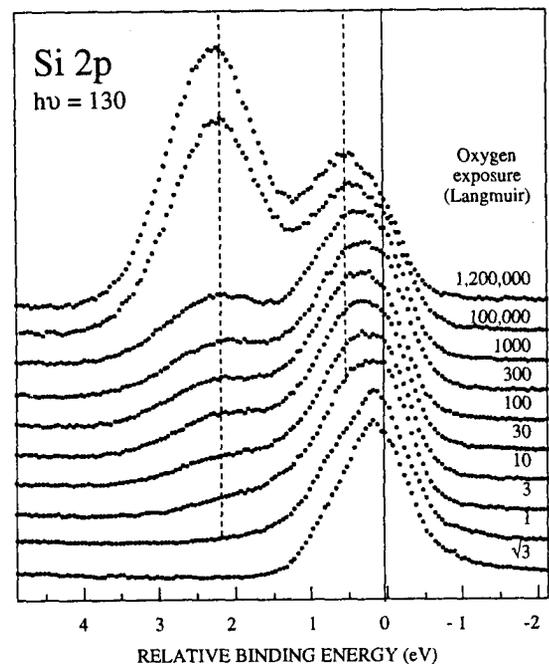
The presence of one sub-oxide ( $\text{Si}^{+1}$ ) besides the  $\text{Si}^{+4}$  from  $\text{SiO}_2$  was recently revealed [1] after in situ oxygen exposures in the  $10^6$  L (1 Langmuir =  $10^{-6}$  torr sec.) range on the  $\sqrt{3}\times\sqrt{3}$  R30° reconstructed surface of 4H-SiC(0001) crystals. The crystals were kept at elevated temperature, from 650 to 950° C, during the oxygen exposures. In studies of initial oxidation of 3x3 reconstructed surfaces of SiC (0001) the presence of three sub-oxides ( $\text{Si}^{+1}$ ,  $\text{Si}^{+2}$ , and  $\text{Si}^{+3}$ ) besides the  $\text{Si}^{+4}$  from  $\text{SiO}_2$  were recently reported [2,3]. These differences motivated us to make a detailed study of the effects induced on the  $\sqrt{3}\times\sqrt{3}$  R30° surface of SiC (0001) after oxygen exposures from the 1 L range up to the  $10^6$  L range. Only two oxidation states,  $\text{Si}^{+1}$  and  $\text{Si}^{+4}$ , can be observed as discussed and illustrated below.

The Si 2p spectrum recorded from the clean  $\sqrt{3}\times\sqrt{3}$  R30° surface and after an oxygen exposure of 1000 L is shown in Fig. 1a. Also shown in the figure is the difference spectrum constructed between the exposed and the clean surface. The  $\text{SiO}_2$  component is clearly seen in the spectrum recorded from the exposed surface and exhibits an energy shift of ca. 2.2 eV. That an additional  $\text{Si}^{+1}$  component is present on the exposed surface is clearly demonstrated by the difference spectrum although it overlaps quite strongly with the bulk SiC component. In Fig. 1b a peak fit procedure has been used to extract the Si 2p components involved and to determine their energy separations. The shift extracted for the  $\text{Si}^{+1}$  component is ca. 0.5 eV. Si 2p spectra recorded after different oxygen exposures are shown in Fig. 2. From these, one can directly see how the two shifted components develop with the amount of oxygen exposure. The vertical dashed lines in the figure are guides to the eye to point out the energy locations of the  $\text{Si}^{+1}$  and  $\text{Si}^{+4}$  components.

The results presented in Figs. 1 and 2 show that only two oxidation states, exhibiting energy shifts of ca 2.2 and 0.5 eV, do develop on the SiC (0001) surface after oxygen exposures at an elevated temperature. Similar results concerning energy shift and number of shifted components were however also obtained when keeping the SiC crystal at room temperature or at liquid nitrogen temperature. The same energy shift for the  $\text{Si}^{+4}$  component was also determined on samples dry oxidized ex situ in a furnace and studied using a higher photon energy [4]. Our results are in contrast to the recently reported findings on the 3x3 reconstruction of SiC (0001) where all four oxidation states were indicated. That four oxidation states do appear on oxidized surfaces of Si is well known [5] and the energy shifts for these oxidation states have been accurately determined. The 3x3 reconstruction is built up of three Si layers [3] on top of the first Si-C bi-layer so that initial oxidation of this surface more resembles that of Si surfaces is therefore not surprising. The above results will be presented and discussed.



**Fig.1.** a) Si 2p spectra recorded at  $h\nu = 130$  eV from the clean  $\sqrt{3}\times\sqrt{3}$  R30° reconstructed 4H-SiC(0001) surface and after oxidation to a total oxygen exposure of 1000 L at a substrate temperature of 800°C. The difference spectrum between after and before oxidation is shown by the bottom curve. b) The results obtained when applying a curve fitting procedure to the 1000 L spectrum in Fig. 1a showing the two oxidation states present.



**Fig.2.** Si2p spectrum recorded after different oxygen exposures onto 4H-SiC at elevated temperature.

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**A high resolution photoemission study of hydrogen terminated 6H-SiC surfaces**

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We report on highly resolved photoemission measurements of hydrogenated 6H-SiC(0001) and (000 $\bar{1}$ ) surfaces. Si2p and C1s core level spectra as well as valence band spectra were recorded at the synchrotron facility BESSY II with an overall energy resolution of about 250meV.

Before hydrogenation the samples (n-type 6H-SiC from CREE, Inc. and SiCrystal) were treated using a four step wet-chemical cleaning procedure. [1] The hydrogenation was carried out by heating the crystals in  $1 \times 10^5$  Pa of ultra-pure hydrogen (8.0) at temperatures of around 1000 °C. [2] In order to avoid contamination, the so prepared samples were transported under UHV conditions ( $p < 1 \times 10^{-6}$  Pa) to the synchrotron facility BESSY II.

The Si2p and C1s core levels of the Si-terminated 6H-SiC(0001) surface after thermal hydrogen treatment are depicted in Figs. 1(a) and 1(b) for surface sensitive ( $h\nu=170\text{eV}$ ) and bulk sensitive ( $h\nu=350\text{eV}$ ) measurements. In the Si2p core level spectrum no chemically shifted component is observable. This is not surprising, because the chemical shift of the Si 2p core level expected on account of the partially ionic Si-H bond is estimated to be 50 meV based on the Pauling electronegativities of the three elements hydrogen, silicon, and carbon.

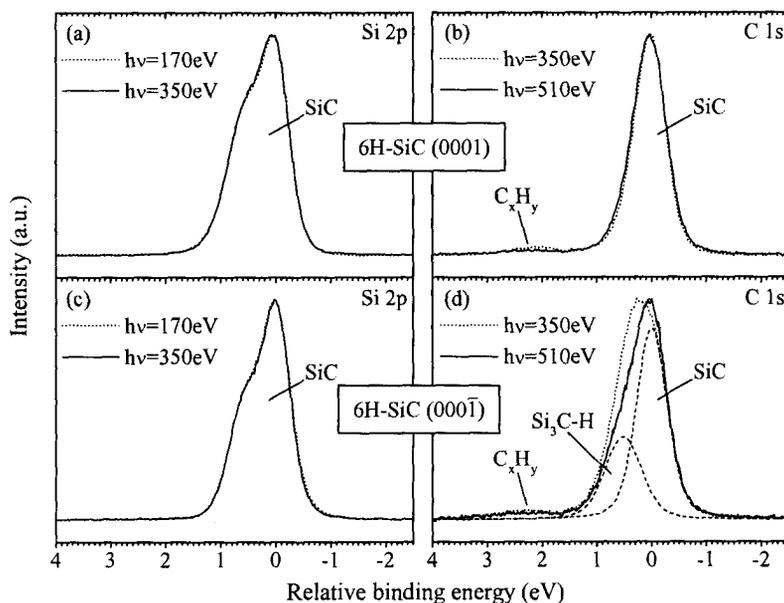


Fig. 1) Si2p and C1s core level spectra of H-terminated SiC(0001) ((a) and (b)) and H-terminated 6H-SiC(000 $\bar{1}$ ) ((c) and (d)). The solid lines correspond to bulk sensitive, the dotted lines to surface sensitive measurements. The dashed lines in spectrum (d) show a deconvolution in two Voigt lines for the bulk sensitive measurement ( $h\nu=510\text{eV}$ ).

This is too small to be resolved by our present resolution. The C1s core level shows an extremely weak chemically shifted component at 2.1 eV higher binding energy which is ascribed to weakly bonded hydrocarbons  $C_xH_y$ . An estimate of the hydrocarbon layer thickness yields  $(0.3 \pm 0.1) \text{ \AA}$  when an effective sampling depth of  $\lambda_{\text{eff}}=4 \text{ \AA}$  is applied for the chosen geometry. The  $C_xH_y$  contamination originates from the eight hours transport to the synchrotron facility BESSY II. The main C1s component and the single Si2p line are attributed to stoichiometric SiC.

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In Fig. 1(c) and (d) the corresponding Si2p and C1s core level spectra of a C-terminated and hydrogenated 6H-SiC(000 $\bar{1}$ ) surface are shown. Again, the Si2p core level does not show chemically shifted surface components. On the other hand, in contrast to the Si-face, the C1s core level exhibits a chemically shifted surface component with 0.5 eV higher binding energy compared to the bulk component. Since hydrogenated surfaces are unreconstructed and no atomic species other than silicon and carbon are detected by XPS the shifted component is ascribed to C-H bonds in a Si<sub>3</sub>C-H configuration. This assignment agrees well with the qualitative argument that hydrogen with a larger Pauling electronegativity than silicon allows a smaller charge transfer towards carbon than silicon. This results in a higher binding energy of carbon in a Si<sub>3</sub>C-H environment in comparison to a Si<sub>3</sub>C-Si environment. The chemically shifted component at 2.1 eV higher binding energy relative to the bulk line is again due to hydrocarbon adsorbates.

Normal emission valence band spectra of hydrogenated SiC(0001) and SiC(000 $\bar{1}$ ) are depicted in Fig. 2. Spectrum (a) was taken on a SiC(0001) surface directly after hydrogenation. Spectra (b) and (c) were recorded on hydrogenated SiC(0001) and SiC(000 $\bar{1}$ ), respectively, after the samples had been exposed to a high dose of synchrotron radiation at  $h\nu=170$  eV. A (1 $\times$ 1) LEED pattern was observed for all three surfaces. We emphasize the dangling bond state D at 0.7 eV above the valence band maximum (VBM) and 1.7 eV below  $E_F$ , which is observed for both terminations after irradiation. The formation of the dangling bond state D is due to radiation induced desorption of hydrogen. Since the dangling bond state is energetically located in the fundamental gap of 6H-SiC below  $E_F$  the bare, unreconstructed silicon carbide surface

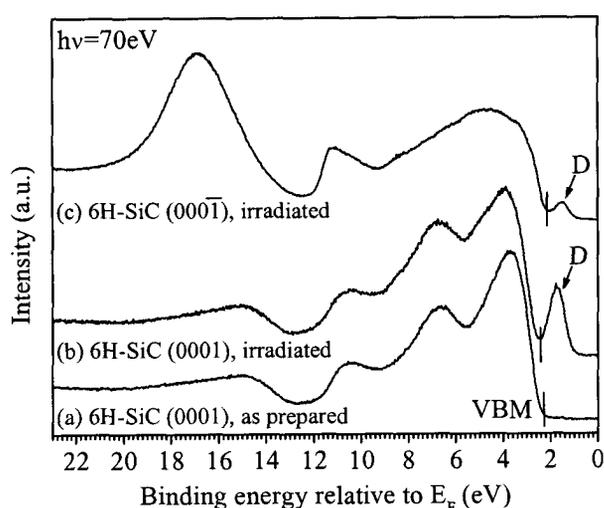


Fig. 2) Valence band spectra of H-terminated SiC(0001) and SiC(000 $\bar{1}$ ) after different preparation steps. VBM denotes the valence band maxima and D the emission maxima due to dangling bonds.

is semi-conducting. This observation is in contrast to theoretical calculations within the one-particle picture which predict metallic behaviour of SiC(0001) and SiC(000 $\bar{1}$ ) surfaces. [3] Similar dangling bond states were observed earlier for 6H-SiC(000 $\bar{1}$ ) with silicate adlayer reconstruction [4] and for SiC(0001) with ( $\sqrt{3}\times\sqrt{3}$ )R30 $^\circ$  reconstruction with a Si-adatom in T4 position. [5] In both cases the dangling bond state lies in the gap below  $E_F$  and the surfaces are semiconducting. This was explained using a Mott-Hubbard description for the dangling bond state which might also be an adequate approach for the clean hexagonal SiC surface.

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## In-situ analysis of thermal oxidation on H-terminated 4H-SiC surfaces

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SiC power MOSFET is the promising for use in high-voltage and low-loss devices. However, details of the structure near the SiO<sub>2</sub>/SiC interface formed by thermal oxidation are not fully understood. To date, many studies have been performed on SiC surface and SiO<sub>2</sub>/SiC interface. Recently N. Sieber et. al. reported that they successfully removed the contaminants are form SiC surface by H<sub>2</sub> annealing and that hydrogenated SiC(0001) and SiC(000-1) surfaces are electronically and chemically passivated with the surface Fermi level position at the bulk position [1]. In this study, the initial oxidation process on a H-terminated 4H-SiC surface achieved by annealing in H<sub>2</sub> was investigated using in-situ Fourier-transformed infrared reflection absorption spectroscopy (RAS), Auger electron spectroscopy (AES), and low energy electron diffraction (LEED), equipped in UHV chambers.

All experiments were carried out in the UHV chamber. Therefore, we can analyze the oxide/SiC interface without effect of contamination except for the prior to the initial loading after chemical cleaning. Commercially available n-type 4H-SiC(0001) Si-face 8° off-axis wafers purchased from CREE Research Inc. were used in the experiments. H<sub>2</sub> annealing of the SiC surface was carried out for 1 hour at 1000°C in 750 Torr of pure hydrogen (the dew point was -83°C). The thermal dry oxidation was performed by heating a H-terminated SiC surface at 400°C in 750 Torr of dry oxygen (the dew point was -108°C) in same UHV chamber. An infrared heating system was employed for the heating samples in the UHV chamber.

AES spectrum for HF treated surface is shown in Fig.1 (a) and that for surface annealed in H<sub>2</sub> at 1000°C is shown in Fig.1 (b). By annealing in H<sub>2</sub>, Auger peak to peak height ratio of C(KLL) to Si(LVV), and that of O(KLL) to Si(LVV) decreased. It is indicated that hydrocarbon and oxide remained on SiC surface were reduced by annealing in H<sub>2</sub>, although they were remained after HF treatment. Fig. 2 shows LEED pattern observed for a 4H-SiC surface immediately after H<sub>2</sub> annealing with primary energy E<sub>p</sub>=150 eV. The diffraction pattern corresponds to a SiC 1×1 surface structure. The spots of the LEED pattern become remarkably sharper and brighter than that for ex-situ HF-treated surface. It is suggested that SiC (1×1) ideal surface is formed by H<sub>2</sub> annealing. From LEED and AES results, SiC surface was cleaning by H<sub>2</sub> annealing. Fig. 3 shows RAS spectra of a 4H-SiC surface at Si-H stretching vibration region. Fig. 3 (a) is the spectrum for a 4H-SiC surface immediately after H<sub>2</sub> annealing at 1000°C. A sharp absorption band at ~2130 cm<sup>-1</sup> was dominantly observed.

It implies that C<sub>3</sub>Si-H bond is formed on a 4H-SiC surface by H<sub>2</sub> annealing at 1000°C. The Fig 3 (b) and (c) show the spectra that thermal oxidation was performed on H-terminated surface in O<sub>2</sub> ambient (750 Torr) at 400°C. The oxidation time of spectra (b) and (c) is 50 and 100 minutes, respectively. The Fig 3 (b) and (c) can be deconvoluted into three components as shown in Fig. 3. The peak frequencies were ~2130, ~2180, and ~2220 cm<sup>-1</sup>. This result indicates that other bonds were formed by oxidation on the H-terminated 4H-SiC surface. It is known that the Si-H stretching frequency shifts toward a higher frequency with the increasing sum of the electron negativity of the atoms or groups bonded to the Si atom. Lucovsky [2] calculated that the stretching frequencies of various Si-H bonds, with O and C are bonded to the Si atoms, that is, ~2180 cm<sup>-1</sup> for OC<sub>2</sub>Si-H, and ~2220 cm<sup>-1</sup> for O<sub>2</sub>CSi-H. These results imply that the oxidation of the Si-C back bond occurred by heating at 400°C in 750 Torr O<sub>2</sub>.

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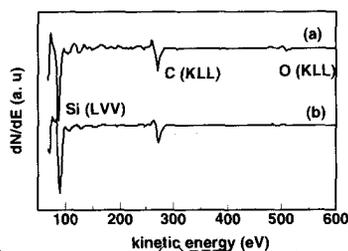


Fig. 1 AES spectra : (a)HF-treatment, (b) annealed in H<sub>2</sub> Fig. 2 LEED pattern for a 4H-SiC surface after annealing

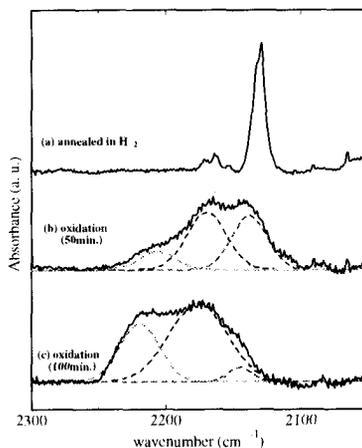


Fig. 3 FTIR-RAS spectra.  
 (a) annealed in H<sub>2</sub>  
 (b) oxidation for 50min.  
 (c) oxidation for 100min.

## Selective Epitaxial Growth of Pyramidal 3C-SiC on Patterned Si Substrate

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Cubic silicon carbide has been grown epitaxially on Si substrates for many years. The heteroepitaxial growth of 3C-SiC on Si has indicated the promise of high mobility devices. However, a high density of interfacial defects (misfit dislocations, voids) as well as other defects (threading dislocations, twins, stacking faults) result in the growth of lower quality material. A suitable approach towards solving this problem is the use of selective epitaxial growth on patterned silicon substrates.

Figure 1 shows the schematic of the fabrication procedure for pyramidal growth. All samples were grown by atmospheric-pressure chemical vapor deposition (APCVD) using hexamethyldisilane (HMDS). The substrates used were a (111)-oriented silicon substrates previously patterned by depositing a SiO<sub>2</sub> layer as the mask, followed by conventional photolithography techniques. The windows are of different shapes (square, circle, hexagonal, parallel lines) with their edges oriented mostly along the <110> directions. Thin 3C-SiC layer was grown on Si exposed through windows. After removing SiO<sub>2</sub> mask, patterning of the seed 3C-SiC layer was achieved by etching process to remove Si around thin 3C-SiC layer. The regrowth was carried out at the growth temperature of 1350°C. As a result of regrowth, selective growth of 3C-SiC pyramid with three facets was observed as shown in Figure 2. This approach prevents the propagation of threading dislocations originating from the 3C-SiC/Si interfaces. Therefore, lateral growth of a 3C-SiC layer until coalescence results in a 3C-SiC layer of low defect-density material. The triangular pyramids were formed on the seed 3C-SiC of different shapes (square, circle, hexagonal) without the shape of parallel lines. This indicates that the facets of each triangular pyramid do not depend on shape, wide and periodicity of the seed 3C-SiC. The air-gap was observed under the free-standing laterally grown 3C-SiC in the cross-sectional SEM.

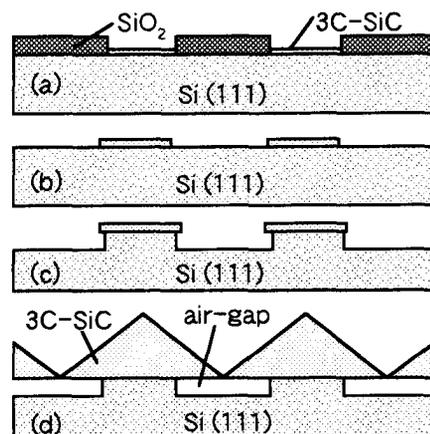


Fig. 1. Schematic of the procedure for pyramidal growth. (a) CVD of thin 3C-SiC layer at window, (b) wet etching of SiO<sub>2</sub> mask, (c) dry etching of Si, and (d) regrowth of 3C-SiC

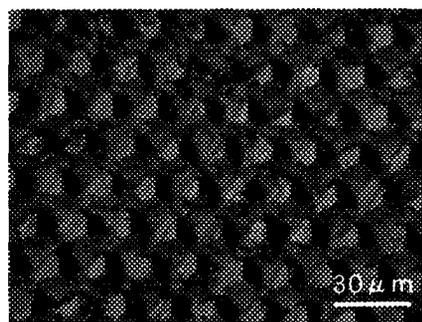


Fig. 2. Nomarski image of a triangular pyramid of 3C-SiC grown selectively by CVD.