Atomic probe microscopy of 3C SiC films grown on 6H SiC substrates

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The surface of 3C SiC films grown on 6H SiC substrates has been studied by atomic probe microscopy in air. Atomic-scale images of the 3C SiC surface have been obtained by scanning tunneling microscopy (STM). STM has confirmed the ⟨111⟩ orientation of the cubic 3C layer grown on the {0001} surface of the hexagonal 6H substrate. The nearest-neighbor atomic spacing for the 3C layer has been measured to be 3.29 ± 0.2 Å, which is within 7% of the bulk value. Shallow terraces in the 3C layer have been observed by STM to separate regions of very smooth growth in the vicinity of the 3C nucleation point from considerably rougher 3C surface regions. These terraces are oriented at right angles to the growth direction. Atomic force microscopy has been used to study etch pits present on the 6H substrate due to high temperature HCl cleaning prior to chemical vapor deposition growth of the 3C layer. The etch pits have hexagonal symmetry and vary in depth from 50 nm to 1 μm.

Silicon carbide is rapidly making progress as an attractive semiconductor material for applications requiring a wide energy band gap and/or operation under extremes of temperature, radiation, power, and frequency. Cubic 3C SiC has been grown[1-4] on Si substrates primarily by chemical vapor deposition (CVD). This enables the construction of devices which combine the wide band-gap semicondutor properties of SiC with the well-developed technology of Si, such as the SiC-Si wide-gap emitter heterojunction bipolar transistor.[5,6] Other applications require an all-SiC device structure, which is normally accomplished by the CVD growth of epitaxial 6H or 3C SiC layers on 6H SiC substrates.[7-11]

It is very important to learn the structure and morphology of the SiC epilayer, in order to understand the growth process as well as to control subsequent device characteristics. Scanning tunneling microscopy (STM) has been successfully utilized[12,13] to provide information on the SiC surface. We have recently[14] reported the first atomic-scale SiC surface investigation, using STM on the surface of 3C SiC grown on {100} Si substrates. The SiC surface, examined in air, displayed an unreconstructed (100) symmetry. In this letter, we report the results of an investigation of the 3C SiC surface grown on {0001} 6H SiC substrates using STM and atomic force microscopy (AFM).

The epitaxial layers investigated were grown at NASA Lewis on vicinal {0001} 6H SiC wafers obtained from Cree Research, Inc. Si-terminated substrates with tilt angles of 0.2° and 0.5° off (0001) were used. The substrates were *n* type with a resistivity of 0.05 to 0.1 Ω cm. The epitaxial growth[15] was carried out at atmospheric pressure with a rf-heated susceptor for approximately 1 h. A pregrowth HCl etch was done for 2 or 30 min at either 1200 or 1350 °C. SiC film growth with silane and propane precursors was performed at 1450 °C, resulting in a growth rate of ~3 μm/h. Growth on substrates etched at 1200 °C resulted in 3C film growth. Grooves were cut in the growth surface of some substrates, producing 1 mm^2 mesas on which 3C films were grown.[15]

SiC surface preparation for atomic probe microscopy consisted of ultrasonic cleaning in acetone, rinsing in methanol, a HF dip, and rinsing in deionized water. No polishing or oxidation of the SiC substrate or epilayer was employed. STM and AFM were performed at the University of Cincinnati using a Digital Instrument Nanoscope II microscope. The STM images were obtained using a PtIr tip biased at ~1 V and a tunneling current of ~2 nA. The STM was calibrated by measuring the highly ordered and well-known surface of pyrolytic graphite.

The surface of the 3C-on-6H SiC layer, as examined by scanning electron microscopy as well as STM, was considerably rougher than that obtained for either very thin 3C SiC-on-Si films grown by rapid thermal CVD or thicker 3C SiC-on-Si films which were polished and oxidized. This produced difficulties in obtaining high resolution STM images over large areas. However, in selected regions which were sufficiently flat and defect free, we successfully imaged the 3C-on-6H surface with atomic-scale resolution. A

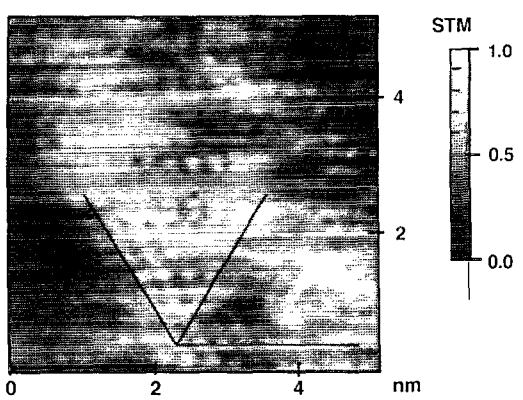

FIG. 1. STM plan-view image of 6×6 nm^2 area of the 3C-on-6H SiC surface.

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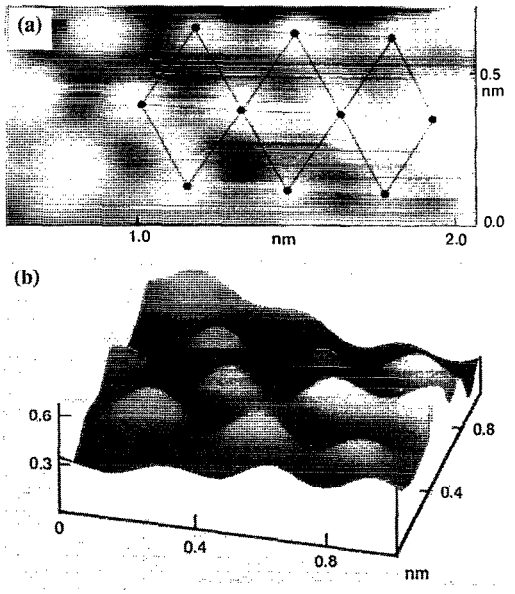

FIG. 2. STM images of 1×1 nm^2 area of the 3C-on-6H SiC surface: (a) plan-view; (b) tilt-view.

plan-view STM image of a 6×6 nm^2 3C SiC area is shown in Fig. 1. Rows of atoms intercepting at an angle of $\sim60°$ are observed, as expected for the $\langle111\rangle$ orientation of the cubic SiC grown on a substrate with hexagonal structure. The sixfold symmetry of the (111) plane of the 3C layer is clearly seen in Fig. 2(a). Here atoms contained in a 1×1 nm^2 area of the STM image are noted to be in good agreement with a superimposed ball-and-stick model of the (111) SiC surface. A tilt-view of the STM image, shown in Fig. 2(b), provides three-dimensional information of the SiC surface. The surface interatomic spacing was measured to be 3.29 ± 0.2 Å, vs the bulk value of 3.08 Å. This discrepancy of 6.8% is probably due to the relatively small number of atoms over which we were able to measure the interatomic spacing with some confidence.

The flat and uniform 3C SiC regions were found in the general vicinity of the edges and corners formed by the grooves cut in the 6H SiC substrate. Flat regions were frequently found to terminate in a series of terraces leading to a much rougher 3C growth region. An example is shown in the STM image of Fig. 3 for a 3C film grown on a 6H

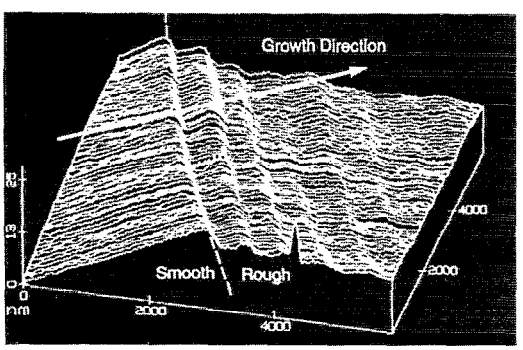

FIG. 3. STM image of growth terraces on the 3C-on-6H SiC film. Arrow indicates growth direction.

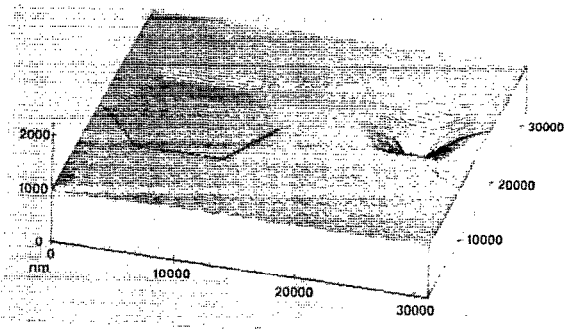

FIG. 4. AFM image of etch pits formed on the 6H SiC substrate after HCl etching at 1350 °C.

substrate with an orientation 0.2° off {0001}. The arrow in Fig. 4 indicates the direction of growth. The tilt from the horizontal of the flat region is 0.35°, while the angle from the horizontal of the first terrace is 0.72°. The vertical drop of the terrace is 4.65 nm, while its horizontal extent is considerably larger, ~368 nm. The terraces displayed in Fig. 3 may represent the agglomeration of atomic steps during the SiC growth process.

We have also investigated the 6H SiC surface using STM. We achieved atomic-scale resolution only on the as-received Si-face 6H substrate, but were unable to discern anything but a random arrangement of atoms. The surfaces of 6H samples which experienced high temperature etching or growth were too rough for observation of individual atoms. The surface of the 6H SiC exposed to HCl pregrowth etching at 1350 °C for 30 min exhibited numerous hexagonal etch pits. We investigated these etch pits using AFM. They vary in depth from ~50 nm to ~1 μm. The walls of the shallow pits have an inclination angle of 4°–6°. The deeper pits have the same initial inclination angle followed by a steeper angle of $\sim15°$ after a depth of ~0.5 μm has been reached. An example containing both shallow and deep etch pits is shown in the AFM image of Fig. 4.

In summary, we have used atomic probe microscopy techniques to investigate the surfaces of 3C and 6H SiC films and 6H substrates. Using STM we have obtained the first atomic-scale image of the (111) cubic SiC film grown on the {0001} hexagonal SiC substrate. STM has also revealed shallow terraces in the 3C SiC surface, perpendicular to the growth direction. Hexagonal pits due to pregrowth high temperature HCl etching have been studied by AFM.

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