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Structural reorganisation of vicinal surfaces on 6H-SiC(0001) induced by hot hydrogen etching

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Abstract

An extended set of vicinal surfaces has been prepared in a 6H-SiC(0001) substrate by mechanically grinding a concave-shaped surface, followed by hot hydrogen etching. During grinding, the different crystallographic planes building up the 6H-SiC polytype are cut under continuously changing polar angles in all azimuthal directions. The local structural reorganisation under hot hydrogen etching is studied by scanning electron microscopy (SEM). The etching conditions for silicon carbide concave-shaped surfaces with vicinal orientations close to (0001) are investigated. Results of hydrogen etching for substrate temperatures of 1700 and 1800 °C are presented. Two types of local bond environments are created, leading to a strong anisotropy of the hydrogen etching. Stable step alignments are observed along the $(11\bar{2}0)$ crystallographic directions, which reflect the symmetry of the bonding of the material. The polar variations of the surface orientation within the concave-shaped surface leads to a variation of the terrace widths with smaller terraces obtained for larger polar misfit.

1. Introduction

Silicon carbide has very interesting electronic, structural and mechanical properties and has become an important semiconductor material. Its crystallography is complex. Silicon carbide crystallises as a polytype with more than 200 different variations. The stacking sequence of the hexagonal bilayers defines each polytype. A frequently used substrate is the 6H-type. Vicinal silicon carbide surfaces, so-called off-axis 6H-SiC substrates, are increasingly used for device fabrication [1]. Silicon carbide films can also be grown with different crystallographic structures (polytypes) where each polytype is accompanied by specific electronic properties. A large spread in the bad-gap energies ranging from, e.g. 2.39 eV for the zinc blende 3C-SiC type to 3.33 eV for the wurtzite structured 2H-SiC type has been measured [1]. The polytype growth is influenced by the surface orientation (vicinality) of the SiC substrate. Changing the vicinality locally on the same substrate allows the growth of different SiC polytypes with electronic properties, which are determined by the polytype. A detailed understanding of the role of vicinal silicon carbide surfaces for the growth of SiC

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films is of great importance for both, fundamental aspects of SiC growth and for technological applications [2]. For example, the growth of nitrides like GaN on vicinal SiC surfaces can be performed without the formation of detrimental screw-dislocations [3]. The control of the substrate step density by adjusting the vicinal surface orientation allows the growth of SiC films with one defined crystallographic structure (single polytype) via the so called ''step controlled epitaxy'' [4].

In previous experiments on silicon, a universal sample for the study of vicinal surfaces has been obtained, by transforming concave-shaped surfaces into an extended set of vicinals through thermal treatment [5,6]. In the case of Si(111) these curved surfaces split up into terraces. Surface morphology and step orientation reflect the threefold symmetry of the crystal. In extension of our previous work on Si, our objective is to investigate the influence of a locally varying surface orientation of a SiC sample on the final morphology after hot hydrogen etching. In preliminary experiments [7] we have observed the morphological reorganisation of concave-shaped surfaces due to hydrogen etching at 1700 °C. The resulting morphology was still rough, with a preferential step alignment in $(11\bar{2}0)$ directions. Here, we present successful hydrogen etching experiments which lead to a regular and smooth structural reorganisations of the initially rough surfaces.

2. Experimental

The samples (4 mm × 15 mm) were cut from an on-axis, nitrogen doped, n-type (resistivity 0.03–0.12 Ω cm) 6H-SiC(0001) wafer [8]. A concave-shaped surface depression was created in the middle of the sample by a dimple grinder, using diamond paste with a grain size of 3 μm. Grinding was stopped when a depth of about 30 μm was obtained in the middle of the dimple. The diameter of the dimple was about 1.5 mm, as checked by scanning electron microscopy (SEM). For the given geometry, the in-plane azimuthal angle φ varied for all samples from $0 < \varphi < 360^\circ$ and the out-of-plane polar angle θ varied for the analysed samples from 0° in the middle of the dimple to about maximum 2–4° as determined through geometrical considerations. Prior to hydrogen (H_2) etching, the samples were cleaned in an ultrasonic bath of trichlorethylene, then acetone, and finally methanol. H_2 etching was performed in a horizontal graphite hot-wall chemical vapour deposition (CVD) reactor [9] at an H_2 pressure of 13 mbar. The etching temperature was varied between 1700 and 1800 °C, and the etching time, i.e. the time of exposure of the sample at high temperature to H_2 was varied between 20 min and 1 h. The resulting structural reorganization is compared to our previous experiments, which were performed at an etching temperature of 1700 °C and equal etching times [7]. The etched samples were subsequently characterised by SEM. The effect of hydrogen etching on the various SiC surface orientations as prepared by dimple grinding was investigated. For comparison, SEM images were also taken on the flat parts of the SiC substrate outside the dimple area.

3. Results and discussion

A concave-shaped surface was obtained on a flat 6H-SiC(0001) sample by dimple grinding. Fig. 1 shows a schematic view of a typical sample. Images were taken on different areas on the concave-shaped surface within the dimple area and, for comparison, also on the adjacent flat parts outside the dimple area. The hydrogen etching conditions required to remove the initial roughness on flat SiC surfaces are well known in the literature [10,11]. When etched for 30 min at 1550 °C under a hydrogen flux at atmospheric pressure [10] all scratches are removed from the flat parts of the substrate. A well-defined distribution of flat terraces and steps is created. These steps are very straight and their heights is 0.75 or 1.5 nm corresponding to half or a complete lattice distance in the *c*-axis (three or six SiC bilayers).

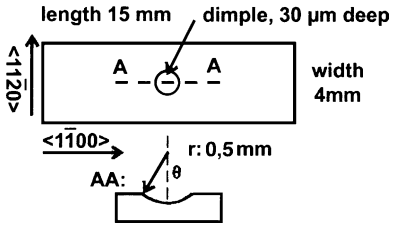

Fig. 1. Sketch of the concave-shaped surface (called ''dimple'') produced in a 6H-SiC(0 0 0 1) surface using a dimple grinder (see text for details).

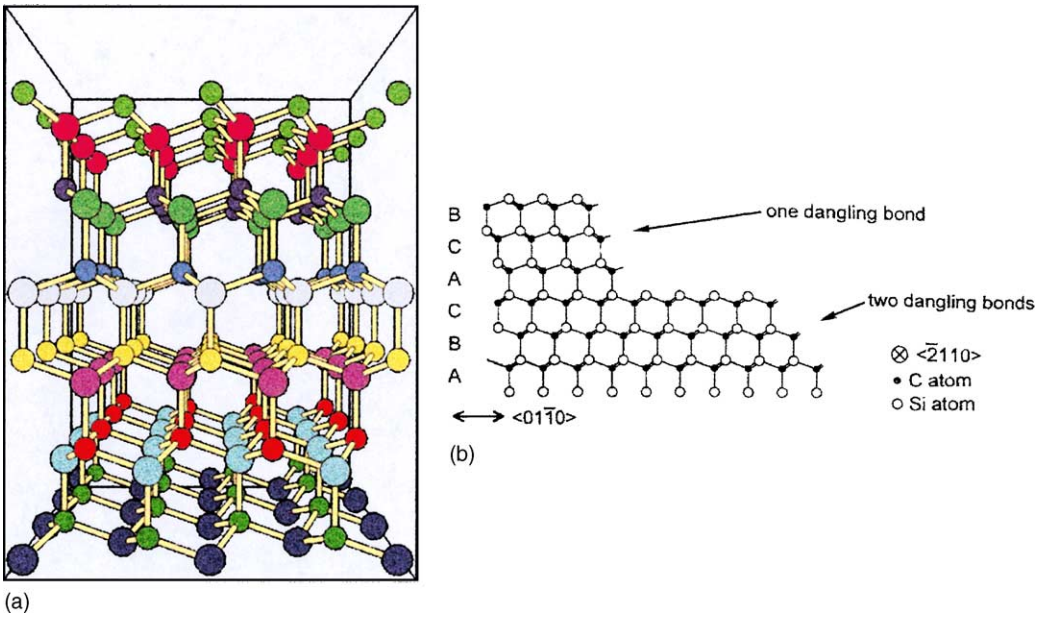

Fig. 2. (a) Crystallographic stacking of the SiC bilayers in a 6H-SiC(0 0 0 1) crystal. (b) Atomic bonds on step risers in the $\{0\,1\,\bar{1}\,0\}$ direction viewed from the $\{\bar{2}\,1\,1\,0\}$ direction. The bond configuration corresponds to steps with three bilayer-height steps. Larger circles stand for Si and smaller circles for C atoms.

However, the etch rate within the dimple area was found to depend on the sample disorientation. On off-axis substrates the etch rate was found to be 30–40% smaller than on on-axis substrates [12]. In the present work, a systematic study of the morphological variations induced through different polar and azimuthal disorientations, was done by analyzing SEM images, which were taken at selected areas within the dimple.

Before we start the discussion of our results, we present in Fig. 2 a sketch of the crystallography of the 6H-SiC(0 0 0 1) polytype of our substrate. Along the *c*-axis of the crystal, i.e. the vertical direction of Fig. 2, six hexagonal SiC bilayers are arranged in two groups of three bilayers each. Within each group, the stacking is hexagonal, but from one group to the other, the lattice is rotated by 60°. As a consequence, the close-packed step edges on 6H-SiC change their nature going from one group to the other. The character of steps running along low index directions alternates and shows either one (SN) or two (SD) dangling bonds (step nomenclature according to Pechman et al. [13]). Fig. 2(b) shows a cross-sectional view along the $\{\bar{2}110\}$ direction. The two groups of step edges are indicated by the number of dangling bonds. The same sequence of two dangling bonds for a cut along the vertical direction in the lower three bilayers versus one dangling bond in the upper three bilayers can also be extracted from Fig. 2(a). The building block of the structure, a fourfold co-ordinated Si atom, is rotated by 60° when going from the lower to the upper half. Therefore, the co-ordination of a Si atom with a C neighbour on the right-hand side changes from two-fold to onefold.

Our results indicate that the hydrogen etching behaviour is strongly influenced by these variations of the local bonding structure. As a consequence, SiC erosion on vicinal surfaces is highly anisotropic. Fig. 3 shows SEM images obtained on equivalent part on the concave-shaped surface. The etching conditions were changed from (a) to (b). The sample in (a) was etched for 60 min at 1700 °C whereas the sample in (b) was etched additionally at 1800 °C for 20 min. The etching conditions used in (a) result in the appearance of a predominance of the most stable step edges, which run along the family of $\{11\bar{2}0\}$ directions, one of the closed-packed step directions. This allows the energetically stable formation of long and straight step edges. Hydrogen etching at a substrate temperature of

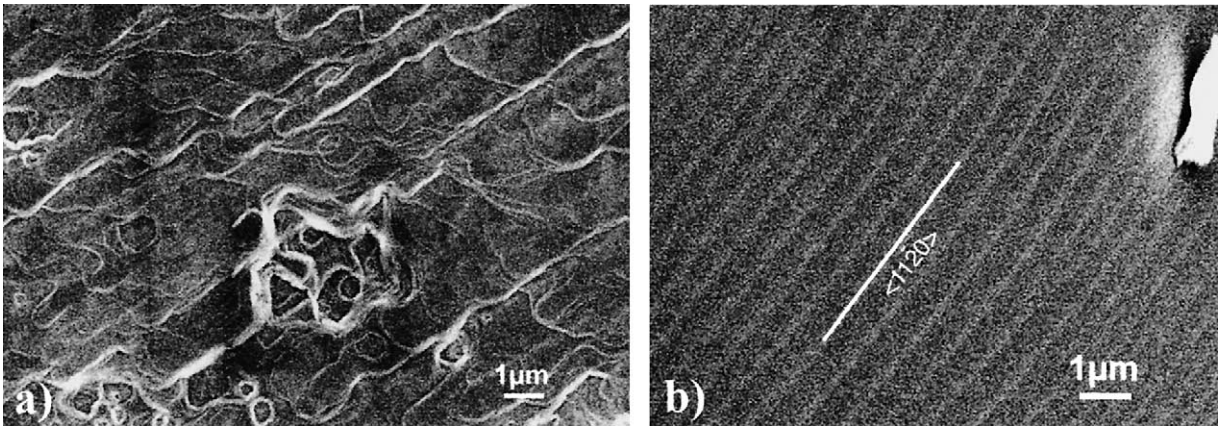

Fig. 3. SEM images of an (a) area on the concave-shaped surface after H_2 erosion for 1 h at 1700 °C; (b) equivalent area on the same sample after an additional H_2 erosion at 1800 °C for 20 min.

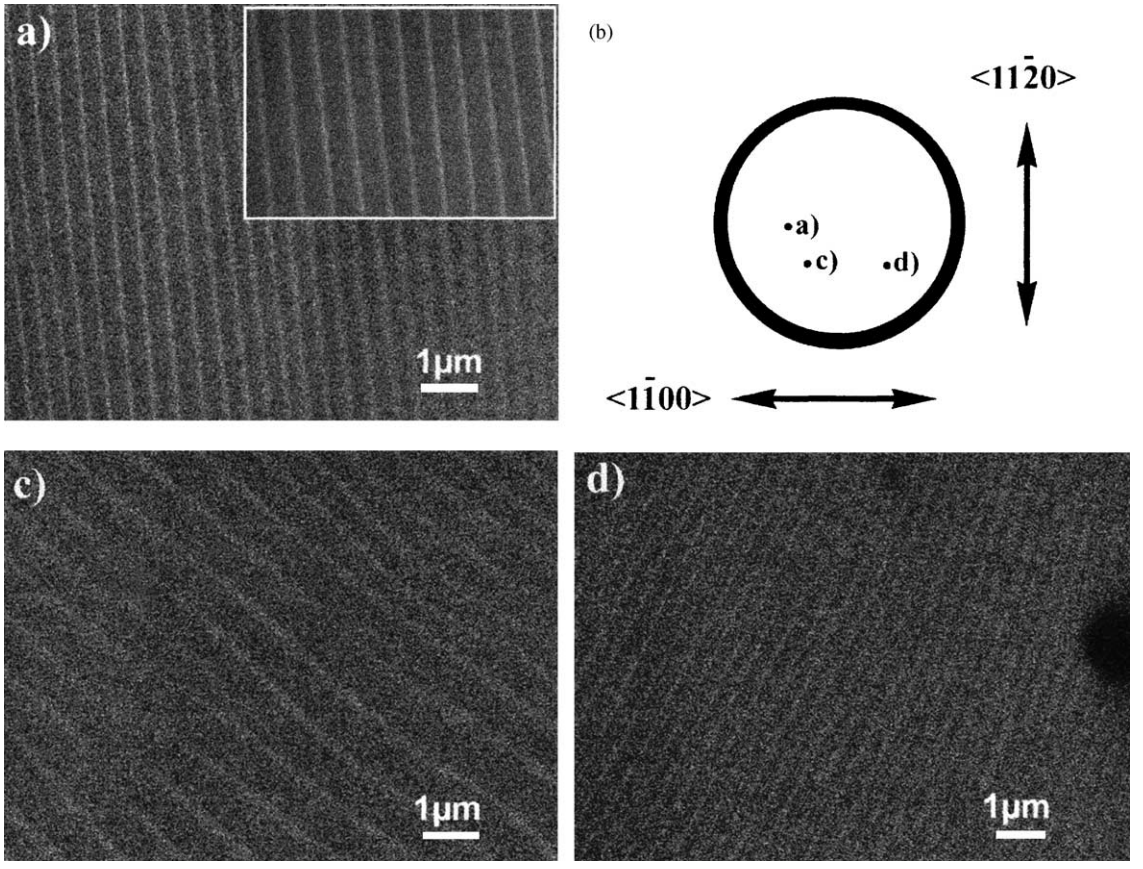

Fig. 4. (a,c,d) SEM images taken on different areas of the reorganised concave-shaped surface, the inset in (a) shows a zoom of the same area. (b) indicates the location of the imaged areas within the dimple.

1800° enables the removal of all rough step bunches, which are still visible in (a), but which are absent in (b). Very straight and regular steps were obtained, which give rise to a very narrow terrace width distribution with a parallel step orientation along $\{1\,1\,\bar{2}\,0\}$. SEM studies have been performed on selected parts of the dimple to study the influence of the azimuthal direction on the structural re-organisation within the concave-shaped surface.. The chosen areas reflect the symmetry of the crystal. Fig. 4(a), (c) and (d) show SEM images of areas as indicated in Fig. 4(b). As already shown in Fig. 3, on all areas a very smooth morphology with very regular, straight step edges was observed. The step direction shows systematic changes depending on the azimuthal misorientation in the dimple. Three different energetically stable alignments can be distinguished. In (a) this direction corresponds to the family of $\{1\,1\,\bar{2}\,0\}$ directions, in (c) and (d) they follow the $\{2\,\bar{1}\,\bar{1}\,0\}$ and $\{\bar{1}\,2\,\bar{1}\,0\}$ directions, respectively. Line scans along the SEM images of the curved surface area at a fixed azimuthal angle give a first, rough analysis of the morphological changes as a function of the polar misorientation. The SEM images shown in Fig. 4 reflect some dependences for small polar angles. In (c) the terrace width is around 1 μm whereas in (d) steps are separated by terraces around 300 nm wide. This larger terrace size in (c) as compared to (b) reflects the smaller polar misfit of area (c) due its closer proximity to the centre of the dimple. More detailed experiments with increased lateral resolution were performed by AFM and will be published elsewhere [14].

4. Summary and outlook

Etching conditions (substrate temperature: 1800 °C, etching time: 20 min, hydrogen flux: 13 mbar) leading to very smooth structural reorganisations of initially rough, curved silicon carbide surfaces were established. First results obtained by SEM show straight step alignments in some distinct, energetically stable directions. Step heights of 1.5 nm corresponding to the height of one unit cell of 6H-SiC were imaged. A more detailed study by scanning tunnelling- and atomic force microscopy of the polar and azimuthal dependencies of the observed step arrangements are in preparation and will be published elsewhere [14].

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