Effect of hydrogen etching on 6H SiC surface morphology studied by reflection high-energy positron diffraction and atomic force microscopy

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Hydrogen-etched 6H SiC (0001) surfaces have been studied by reflection high-energy positron diffraction and atomic force microscopy. It was found that residual damage on the surfaces were effectively removed by the hydrogen etching as compared to the HF etching after the oxidation. The hydrogen-etched surfaces were atomically flat. After the oxidation following the hydrogen etching, the surface roughness was found to increase and an anomalous dip structure appeared in the rocking curve of the reflection high-energy positron diffraction. © *2000 American Institute of Physics.* [S0003-6951(00)02209-9]

Silicon carbide (SiC) is a promising semiconductor as high power, high frequency, high temperature, and radiation-resistant device materials. These are due to the wide band gap, high electron saturation velocity, high breakdown voltage, and high thermal conductivity of SiC over those of Si.[1] To fabricate high performance devices, it is essential to obtain atomically flat, clean, and inactive surfaces without oxides. It is however difficult to remove oxides completely on SiC surfaces and to terminate the dangling bonds with atomic hydrogen by the HF treatment ordinarily used in the Si technology.[2]

The etching of SiC surfaces in hydrogen atmospheres at high temperatures has long been recognized.[3] Recently, it has been found that damage on SiC surfaces formed during slicing and polishing processes are removed by the hydrogen etching above 1400 °C.[4,5] Moreover, dangling bonds on SiC surfaces are found to be terminated with atomic hydrogen after hydrogen etching above 1100 °C.[6] It is important to investigate hydrogen-etched SiC surfaces both in fundamentals and applications.

Reflection high-energy positron diffraction (RHEPD) has the capabilities to analyze surface structure.[7,8] Using the total reflection of positrons at the topmost surface, the sensitivity of diffracted beams to the surface is enhanced as compared to reflection high-energy electron diffraction.[8,9] In this research, we have studied hydrogen-etched 6H SiC surfaces using RHEPD and atomic force microscope (AFM) observation. The results of RHEPD and AFM were compared in detail.

Mirror-like polished Si faces of 6H SiC (0001) were used in this study. After decreasing treatment by successive boiling with acetone, sulphuric acid (H_2SO_4) and aqua regia (HNO_3:HCl=1:3), the specimens were dipped in 5%-HF to remove native oxides and then subjected to the following different treatments: (i) simply boiling for 2 min in ultrapure water (specimen No. 1), (ii) pyrogenic oxidation at 1100 °C for 2 h in order to create a fresh oxide layer, dipping in 5%-HF to remove the oxide layer and boiling in ultrapure water (specimen No. 2), (iii) annealing in H_2 gas (100 Torr) with a flow rate of 2 slm at 1400 °C for 8 h (specimen No. 3), and (iv) oxidation in an O_2 gas at 800 °C for 4 h, dipping in 5%-HF and boiling in ultrapure water after the H_2 annealing (specimen No. 4). The treatment (iv) was made to observe the effect of oxidation on the hydrogen-etched surface. The specimen No. 3 showed almost perfect hydrophobicity, which suggests that oxides on the surface were effectively removed by the hydrogen etching and surface dangling bonds were terminated with atomic hydrogen.[6] The specimens Nos. 1, 2, and 4 did not show perfect hydrophobicity indicating that oxides were not completely removed by the HF treatment. Surface morphology of the above specimens was observed by JEOL JSPM-4200 atomic force microscope. Root mean square roughness (R_q) was also determined. Reflection high-energy positron diffraction measurements have been performed at $[1\bar{1}00]$ incidence using a 20 keV positron beam generated by an electrostatic method described elsewhere.[10] The rocking curves (intensity as a function of glancing angle θ) for specularly reflected positrons were also determined as integrated intensity.

Figures 1(a)–1(d) show the AFM images of the specimens Nos. 1 to 4. It is found that the surface morphology strongly depends on the treatment. From Figs. 1(a) and 1(b), the surfaces of the specimens Nos. 1 and 2 are fairly rough. The size of the bump on specimen No. 2 is slightly larger than that on specimen No. 1. The roughness R_q of the specimens Nos. 1 and 2 is determined to be several tenths of Å and approximately 10 Å, respectively. Thus, the pyrogenic oxidation and subsequent HF dipping reduce roughness. Figure 1(c) shows that the H_2 annealing drastically changes the surface morphology: The bumps observed for the specimens Nos. 1 and 2 disappear and well-ordered wide terraces (approximately 70 nm) appear although unspecified irregularities (white parts) are observed on the terraces. The step height between each terrace is approximately 2 Å that is

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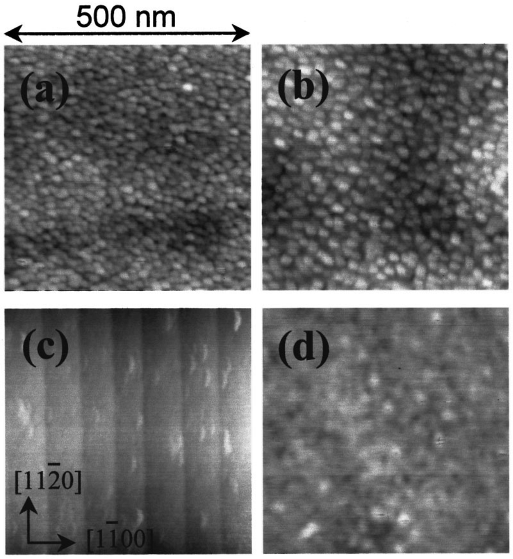

FIG. 1. AFM images observed for (a) the specimen No. 1: boiled in ultrapure water after HF dipping, (b) the specimen No. 2: boiled in ultrapure water after pyrogenic oxidation at 1100 °C for 2 h and subsequent HF dipping, (c) the specimen No. 3: annealed in a H_2 gas (100 Torr) at 1400 °C for 8 h, and (d) the specimen No. 4: boiled in ultrapure water after oxidation in an O_2 gas (1 atom) at 800 °C for 4 h and subsequent HF dipping following the H_2 annealing.

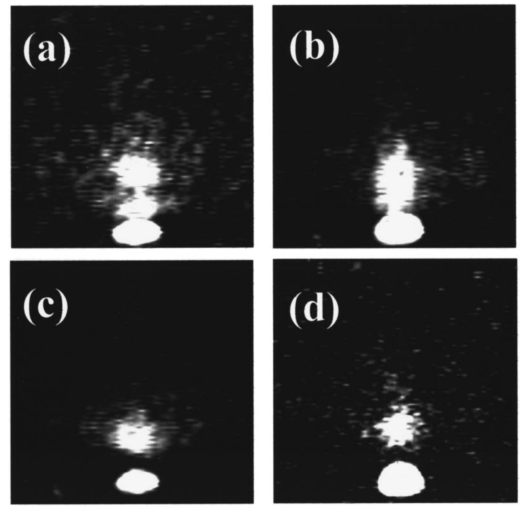

FIG. 2. Specular patterns ([1$\bar{1}$00] incidence) in the total reflection region (~1.2°) from (a) the specimen No. 1: boiled in ultra pure water after HF dipping, (b) the specimen No. 2: boiled in ultrapure water after pyrogenic oxidation at 1100 °C for 2 h and subsequent HF dipping, (c) the specimen No. 3: annealed in a H_2 gas (100 Torr) at 1400 °C for 8 h, and (d) the specimen No. 4: boiled in ultrapure water after oxidation in an O_2 gas (1 atom) at 800 °C for 4 h and subsequent HF dipping following the H_2 annealing. The lowest spots in each picture are direct beams.

comparable to the Si–C bond length (1.87 Å). The roughness R_q in the terrace is less than 1 Å. These results suggest that atomically flat terraces and bilayer steps are formed by the H_2 annealing. As shown in Fig. 1(d), the step structure formed due to the H_2 annealing disappears after the oxidation at 800 °C and HF treatment (specimen No. 4). Probably, steps are eliminated by removing the oxide layer by HF dipping. The roughness R_q of this specimen is 2–3 Å suggesting the flatness after the H_2 annealing is well maintained.

Now we describe the results of RHEPD experiments. In the preliminary experiments, all the specimens exhibited 1×1 diffraction patterns. That is, the surfaces have the bulk-like periodicity. This result is in accordance with the previous low-energy electron diffraction experiments.[11] The appearances of diffraction patterns (i.e., not halo patterns) also indicate that the surfaces are not completely covered by amorphous oxide layers even if oxides remain.

Figures 2(a)–2(d) show the patterns of specularly reflected positrons for the specimens Nos. 1 to 4 surfaces in the total reflection region, i.e., $\theta<1.7°$. Figure 2(a) shows that the specular pattern of the specimen No. 1 definitely splits. Considering the results of the AFM observations, the split pattern is probably due to a scattering of positrons at the edges of the bumps. The specular pattern of the specimen No. 2 is no longer splitting as shown in Fig. 2(b). The differences in the specular patterns of the specimens Nos. 1 and 2 may reflect their different surface morphology shown in the AFM images (Fig. 1). The improvement of the specular pattern for the specimen No. 2 suggests that the surface becomes relatively smooth as compared to that of the specimen No. 1. The streak pattern however indicates the broadening of the Laue function probably due to small terraces on which positrons are scattered. On the contrary, Fig. 2(c) shows that the specimen No. 3 gives a spot like pattern. This result shows that the hydrogen-etched surface is atomically flat as proven by the AFM observations. The specular pattern of the specimen No. 4 is also spot-like as shown in Fig. 2(d). The flatness obtained by the H_2 annealing is not completely lost by subsequent oxidation. This also seems to be consistent with the AFM observations.

Figure 3 shows the rocking curves of specular spots. In Fig. 3(a), the intensity in the high angle region is also shown. The peaks above 1.7° are assigned from the first to the fourth Bragg peaks from the Bragg condition.[12] To see the differences of the rocking curves in detail, the intensities corrected by dividing with the geometrical factor ($\sin\theta$) are shown in the inset of Fig. 3 for the specimens Nos. 1–3. From the curve (a) in the inset, the specular intensity of the specimen No. 1 is somewhat suppressed below 1.3°. The curve (b) for the specimen No. 2 shows a plateau between 0.8° and 1.5° but is still suppressed at lower angles. The positron reflectivity should not decrease in the total reflection region for an ideally flat surface.[8] The suppressed intensities of the specimens Nos. 1 and 2 at low angles are explained as the repelling and/or absorption of incoming and outgoing positrons by large roughness. On the contrary, the curve (c) in the inset shows that the specular intensity of the specimen No. 3 does not decrease in the total reflection region. This result shows that the surface roughness of the specimen No. 3 is much reduced by the hydrogen etching. Thus the change in the specular intensities in the total reflection region from the specimens Nos. 1–3 is explained as the improvement of surface roughness as well as the change in the specular patterns shown in Fig. 2.

As shown above, the roughness of the specimen No. 4 is 2–3 Å at most, which is only slightly larger than that of the specimen No. 3. The specular pattern is spot-like as well as the specimen No. 3 as shown in Fig. 2(d). However, Fig. 3(d) shows that the specimen No. 4 gives rise to an anticipated dip structure at 1.5° in the rocking curve. The dip structures appear due to the interference effects of positron

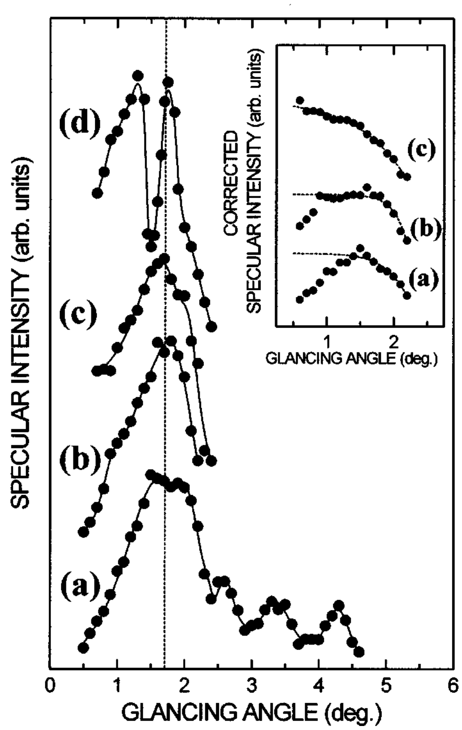

FIG. 3. RHEPD rocking curves of the specular beams ($[1\bar{1}00]$ incidence) for (a) the specimen No. 1: boiled in ultrapure water after HF dipping, (b) the specimen No. 2: boiled in ultrapure water after pyrogenic oxidation at 1100 °C for 2 h and subsequent HF dipping, (c) the specimen No. 3: annealed in a H_2 gas (100 Torr) at 1400 °C for 8 h, and (d) the specimen No. 4: boiled in ultrapure water after oxidation in an O_2 gas (1 atom) at 800 °C for 4 h and subsequent HF dipping following the H_2 annealing. The vertical dotted line shows the critical angle of the total reflection. The inset shows the intensities corrected with the geometrical factor (sin θ) for the specimens Nos. 1–3.

waves caused by adsorption on flat surfaces and/or atomic scale (monolayer and bilayer) roughness on large terraces.[8,13] Considering residual oxygen after HF treatment,[11,14] the above dip structure is possibly caused by oxygen adsorption. An alternative explanation is the roughness formed during oxidation and subsequent HF dipping. It is important to clarify the effect of oxidation on the surface flatness both from fundamental and technological viewpoints. Further studies are in progress to elucidate the origin of the dip in the total reflection region.

In summary, 6H SiC (0001) surfaces have been studied through RHEPD and AFM observations. Both methods provide a consistent result that atomically flat surfaces are obtained by the hydrogen etching. The flatness of the hydrogen-etched SiC surface is well maintained even after oxidation while an anomalous dip appears in the RHEPD rocking curve. The hydrogen etching is expected to be one of the important processes to obtain flat and inactive surfaces in the SiC device fabrication.

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