Spatial Analytical Surface Structure Mapping for Three-dimensional Micro-shaped Si by Micro-beam Reflection High-energy Electron Diffraction

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Spatially arranged surfaces on the micro-rod structure, which was three-dimensionally (3D) architected on a Si(110) substrate have been thoroughly investigated by a system with micro-beam reflection high-energy electron diffraction (μ-RHEED) and scanning electron microscopy (SEM). The combination of μ-RHEED and SEM realized analytical structure investigation of 3D surfaces with the spatial resolution of sub micrometer for the 3D rectangular shaped rod consisting of a (110) top surface (20 μm wide) and {111} vertical side surfaces (10 μm wide). Exhaustive mapping revealed the peculiar reconstructed surface structures: Si(110) “16 × 2” single domain and {35 47 7} facet surfaces locally appeared on the interconnected edge region on the 3D structure in addition to the “16 × 2” and 7 × 7 super structures on flat top (110) and side {111} surfaces, respectively. The formation mechanism for “16 × 2” single-domain structure near the corner edge of the (110) surfaces and {35 47 7} facets on the corner edges between (110) and {111} surfaces were discussed from the viewpoint of the surface stability on the 3D geometrical shaped Si structure.

Keywords Si micro-structures; Reflection high energy electron diffraction; Scanning electron microscopy; Surface structure

I. INTRODUCTION

The scale of transistors has been continuously shrunk year by year in order to develop information technology society. The planar type transistor reached its structural limit and have been replaced with transistors having three-dimensional (3D) structures in novel devices [1, 2]. Such 3D structures are generally fabricated by etching single crystalline Si(001) substrates [3]. The surfaces of the as-manufactured 3D structure were no well-defined and terminated with a hydrogen or oxide layer. Such as-manufactured termination may hide interesting properties of surfaces, which depend on the orientation of the crystal surfaces. To understand the surface properties of a 3D structure, it is elemental to start with a 3D structure surrounded by well-defined surfaces. Since the device performance, typically carrier mobility, is severely affected by surface orientations [3], the arbitrary control of surface structures on the 3D Si nanostructures has been desired for further designing of Si transistor. Comparing two-dimensional (2D) planar Si surfaces easier to be accessed, however, a little attention has been focused on the surface structures on 3D Si by the difficulty of fabricating well-controlled 3D surface and the lack of the investigation technique for the 3D arranged surfaces.

Recently, Hattori et al. have successfully fabricated 3D Si structures by optimizing dry and wet etching conditions, and
created the structures surrounded by atomically-flat clean surfaces such as (001), (111), and (110) after thermal treatments in an ultra-high vacuum (UHV) [4]. For instance, they observed \(7 \times 7\) and \(16 \times 2\) superstructures from the vertical \{111\} side surfaces and horizontal \{110\} surfaces, respectively, of the rectangular micro-rods fabricated on a Si(110) substrate using conventional reflection high-energy electron diffraction (RHEED); the coexistence of \(7 \times 7\) and \(16 \times 2\) patterns was demonstrated using an electron beam of a size of 0.5 mm covering both side and substrate surfaces. The studies are pioneer for the creation and observation of the surface structures of 3D Si structures. However, microscopic observation covering over the 3D structure was not demonstrated yet, which can inform the local structures formed on the side and 2D planar as well as those on the inter-connected edge area of the 3D architectured structures.

Structural deformation by the thermal treatment is another important phenomenon to make a durable 3D structure. To obtain a well-defined clean surface for Si, the thermal treatment close to melting temperature is usually required in UHV condition. Such high temperature treatment must deform a crystal into its equilibrium shape, where the total surface energy is minimized by surrounded by stable surfaces with lower surface energy in the thermodynamical theory [5, 6]. For Si crystal, several low and high index surfaces, such as \{111\}, \{110\}, \{100\}, \{331\}, \{311\}, and \{35 47 7\}, have been confirmed to exist as stable clean surfaces [7–9]. The thermal treatment will degrade the designed 3D structures with sharpen corner edges into those with round edges or facet surfaces depending on the surface orientations.

Indeed, Bermond et al. studied the thermal deformation of Si micro columns by the electron microscope to determine the surface energy [10]. Nakamura et al. observed a significant deformation at relatively low temperature 650°C on Si micro grooves by the UHV scanning electron microscopy (SEM) study [11]. Identifying the Miller index of micro-surfaces by crystallographic methods is important for understanding the deformation.

For the observation of local surfaces in 3D Si structures, it is required the microscopic methodologies evaluating surface orientation and reconstruction over the 3D Si structure with multiple oriented surfaces including facet surfaces. One of the observation methods is scanning probe microscopy (SPM) which can reveal surface morphology and atomic-scale topography. Indeed, using a scanning tunneling microscopy Yang et al. observed a side surface in a 3D Si structure by preparing a special geometry of the sample to a probe tip [12]. However, SPM cannot provide the structure map covered on the 3D structure by steric hindrance. Another method is a combination of electron microscopy and electron diffraction, such as the low energy electron microscopy (LEEM) [13] and micro RHEED (μ-RHEED) by the apparatus of reflection electron microscopy (REM) [14] or SEM [15]. LEEM is, however, not suitable for the present purpose because it basically requires a flat specimen for imaging. The μ-RHEED with REM and SEM can fit to observation of the 3D structures, in which surfaces with different height and different orientation cover the structures, when the precise manipulation of the sample rotation and positioning is provided. However, the freedom of the sample manipulation is usually limited by a short working distance of the electron microscope.

In this study, we have designed a μ-RHEED system for analytical surface structure arrangement mapping on 3D-shaped Si samples; the system is a sub-micrometer-scale microscopy technique for obtaining the RHEED pattern from 3D structures. The micro-beam electron gun having a large working distance enables the simultaneous imaging of the assembled surfaces on the 3D structured sample as SEM images and RHEED patterns, and the identification of the surface structures, that is, surface morphology, surface orientation defined by Miller index, surface ordering, and surface reconstructed structures. We have applied this technique for the 3D-shaped Si sample with \{111\} vertical side surfaces on \{110\} substrate, and successfully observed the diffraction patterns from \{111\} vertical side surfaces, \{110\} horizontal surfaces and, in addition, \{35 47 7\} facet surfaces appeared at the edge between \{110\} horizontal and \{111\} vertical surfaces. Moreover, the appearance of single domain structure of \(16 \times 2\) reconstruction suggested the formation of the monatomic steps running parallel to the corner edges of the 3D structure.

II. EXPERIMENTAL

A. μ-RHEED system

Figure 1(a) shows the schematic illustrations of the μ-RHEED equipped with a Schottky electron gun combined with a scanning electron microscope module (FEI Schottky SEM module), a 3-inch RHEED screen with an MCP intensifier (a gain of ~10³), and a 4-axis sample manipulator. The electron gun can provide a focused beam at a long working distance (WD) up to 190 mm with a typical beam current of a few 10⁻⁹ A. In order to provide a sufficient space for sample manipulation, the WD was set to about 100 mm in the present system. Because the present focused beam has the long WD, the diffraction spot was not as broad as the convergent beam diffraction. The expected beam diameter was about 100 nm at 10 keV, but in our system, despite the installation of a magnetic shield in the chamber, the effective beam size was about 1 μm due to the beam oscillation by the residual magnetic field. Since the electron beam was incident at a glancing angle in RHEED measurements, the footprint of the 1 μm beam on the surface was elongated along the beam direction to 10–30 μm for typical grazing angles (2°–5°). Thus, the longitudinal spatial resolution was significantly degraded depending on the incidence angle, while the transverse resolution along with the surface corresponds to the beam size. The gun was equipped with an electrostatic octupole lens, which enable the raster scan of the beam in the area of 10 mm × 10 mm in sample position.

The μ-electron beam was used for RHEED as well as SEM observations. A RHEED pattern from a selected posi-
tion of sample was displayed on the screen via the MCP intensifier and captured with a CCD camera (1440 × 1216 in pixel size). For SEM observation, the secondary electrons excited by the beam were collected by secondary electron (SE) detector, and a SEM image was obtained when the intensity of SE was displayed along with the raster scan of the beam. The raster scan and signal input were performed using an analog I/O device (NI, USB-6366) controlled by a self-made program in NI LabVIEW. The system was able to show SEM images at eight frames per second with a reasonable resolution.

To investigate arbitrary oriented surfaces of the 3D microstructure by µ-beam, we have built a 4-axis sample manipulator that enables precise positioning and rotations. The long WD of the e-gun allows the specimen to rotate over a wide angle-range in the present system. The sample manipulator was constructed based on a mechanical rotation stage for glancing angle (θ), which can be rotated from outside of the chamber by a mechanical rotary drive. A piezo rotary stage (attocube systems, ECR3030) for azimuth angle (φ) rotation and two piezo linear translation stages (attocube systems, ECS3030) for in-plane (xy) translation are stacked on the θ-stage as shown in Figure 1. Since each piezo stage is equipped with a position or rotary encoder, the sample position and φ angle can be set accurately with an accuracy of 50 nm and 10⁻³ degree, respectively. Using this manipulator, a µ-RHEED pattern at an arbitrary point of the sample with an arbitrary incident angle can be measured. Note the irradiation point was controlled by both the sample manipulator and the e-beam deflection. The manipulator has two electrode contacts that can be used for the resistive heating of the sample.

B. Si 3D microstructures

The microstructures were produced on a commercial mirror polished Si(110) wafer (Sb-doped, 1–10 Ω cm) using a photo-lithographic technique and wet etching. The details of the fabrication were described in Ref. 4. In the present study, a column of rectangular rods with {111} vertical side-surfaces was fabricated on the wafer as shown in Figure 2(a, b). Each rectangular rod was designed to be formed by top (110) surface and side (111)/(111) surfaces, and their width, height, and length were 20, 10, and ~2500 μm, respectively. Hundreds of rods were evenly arranged on the substrate with a period of 60 μm. A crystal stereograph
viewing from [110] direction is shown in Figure 2(c) to explain the crystal orientation of the rods, and the rods were aligned parallel to [112] direction.

The micro-structured wafer was cut into 3 mm × 20 mm × 0.3 mm, and then the sample was chemically cleaned by acetone and ethanol, and rinsed with pure water. After dried by blowing with dry N₂ gas, the sample was clipped on the sample manipulator of the UHV chamber. The sample can be resistively heated on the manipulator in the UHV, and the sample temperature was measured by an optical pyrometer. The sample was carefully degassed by resistive heating up to 1000°C in the UHV condition. To obtain a clean surface, the sample was flashed at 1260°C for 5 s and then annealed at 1000°C for 1 min. The procedure was repeated several times to obtain the clean surface. SEM and RHEED observations were performed on the sample after cooling down to room temperature.

III. RESULTS

Figure 3(a, b) shows typical μ-RHEED patterns observed from the top surface of a rectangle rod. The incident e-beam direction was [112], i.e., along the rod length direction. Both patterns correspond to “16 × 2” superstructure expected for Si(110) clean surface [4]. However, there is a clear difference between the patterns measured at the center part [Figure 3(a)] and the near edge part [Figure 3(b)]. As schematically shown in Figure 3(c), “16 × 2” can take two equivalent orientations, i.e., “16 × 2”-A and “16 × 2”-B, on the (110) surface so that it usually takes double domain (DD) structure, in which the domain A and B are equally distributed [16]. When the experimental patterns are compared with simulated spot positions, which are shown in Figure 3(d), it is clear that both of “16 × 2”-A and “16 × 2”-B patterns were observed in Figure 3(a), but “16 × 2”-A was dominant in Figure 3(b), where the strong spots were observed along 0th-order Laue ring (L₀) and half order ring (L₁/₂) as expected for “16 × 2”-A. This indicated that “16 × 2”-A domains, whose × 2 direction was parallel to [112], dominantly cover the surface close to the corner edge, i.e., the single domain (SD) “16 × 2” was formed.

A typical diffraction pattern from the side surface of the rod and its schematic illustration are shown in Figure 4(a, b). The incident direction was slightly off from [112] in the azimuthal direction. Since the pattern was diffracted from the vertical plane, a vertical shadow edge as well as a horizontal one from the substrate appeared on the screen. The observed pattern is consistent with the simulated 7 × 7 pattern from the vertical plane shown in Figure 4(b). Thus, we succeeded in observing the diffraction patterns from the top (110) surface with a width of 20 μm and the side (111) surface with a width of 10 μm of the Si rectangular rod.

In addition to the patterns from (110) and {111} surfaces, another interesting pattern was observed at the corner of the rod, as shown in Figure 4(c). In this image, in addition to “16 × 2” pattern, there were the streaks that inclined ~11° from (110) were overlapped onto “16 × 2” pattern. The tilted streaks indicate the faceting occurred at the corner edge between (110) and (111) surfaces.

Among the candidates of known stable Si surfaces, (35 47 7) surface has the facet angle of 10.74° from (110), which is close to the observed inclined angle; note that (35 47 7) is slightly (0.8°) off from the [112] zone as shown in the stereograph of Figure 2(c).
The simulated spot positions for (35 47 7) 1 × 1 lattice is overlaid with that of “16 × 2”-A in Figure 4(d). Spots were not clear in the experimental pattern but the most of spots and streaks are well interpreted by (35 47 7) 1 × 1. Therefore, we concluded that (35 47 7) facets were formed at the corner edge between (110) and (111). We have also observed (47 35 7) facets, which are equivalent to (35 47 7), at the counter corner of the rod.

In order to investigate the distribution of surface orientations, we have measured diffraction patterns from 41 points across the rods. While the focused beam was fixed, the sample was moved by the piezo stage with a constant interval of 1.9 μm in horizontal. As shown in the lower part of Figure 5, the sample was scanned from the left edge of the rod to the right edge of the neighbor rod. The 41 patterns can be classified into three types: “16 × 2”-DD, “16 × 2”-SD, and {35 47 7} facet patterns. In order to show an overview of the position dependence, the central vertical region of each pattern was picked up as a ribbon-shape [see the broken rectangular in Figure 3(a)], and compiled into an image, in which the 41 ribbon-shaped patterns were arranged according to the beam position. The compiled image is shown at the upper part of Figure 5, in which the contrast was suppressed around the L0 region to prevent intensity saturation.

The compiled RHEED patterns clearly showed the changes in the diffraction patterns depending on the position over the rods. As indicated in Figure 3(d), “16 × 2”-A shows spots along L0 and L1/2 rings, and “16 × 2”-B shows vertical streaks above and below L0 position. At the center part of the top and the bottom surfaces, both of the L1/2 spots and the vertical streaks were observed, indicating that the double domains co-existed there. On the other hand, as the position approaches from center to the corner edge, for both the top and bottom surfaces, the intensity of streaks decreased, but that of the L1/2 spots increased. This means the DD structure transformed to the SD structure near the corner edge although the boundary between SD and DD was blurred. According to the RHEED observation, SD and DD regions are mapped in blue and light blue, respectively, on the rod surface in Figure 5. The SD regions existed symmetrically with respect to the center of the upper surface and the lower surface, and the width of SD region and DD region was about 6 μm each on the top surface and about 12 μm each on the bottom surface.

The {35 47 7} facet patterns were observed at both the upper and the lower edges of the rod structure as mapped in red on the rod in Figure 5. The width of the facets was about 2 μm at both the top and bottom edges, but the facet should be smoothly connected to the (110) and {111} planes via a curved surface.

**IV. DISCUSSION**

While the two equivalent orientations exist for Si(110) “16 × 2” reconstruction as shown in Figure 3(c), the formation of SD is often reported under certain conditions in previous works [16–20]. There are mainly two approaches to produce the “16 × 2” SD structure. One is the electromigration effect depending on the current direction of heating, and the other is a vicinal orientation of the Si(110) substrate. Yamada et al. suggested that the electromigration induced the SD formation by comparing the current flow effect of two different orientation of rod-shaped Si(110) wafers [17]. They dominantly observed the SD structure on the wafer whose long side, i.e., the current flow direction, was [112]. Conversely, Sakamoto et al. insisted that the direction of the
current did not affect the formation of SD [19], and Lewis et al. reported that SD “16 × 2” could be formed on the vicinal surface with aligned monatomic steps running parallel to [112] [20]. Since the present rectangular rods aligned almost perpendicular to the current flow direction and steps running parallel to [112] are expected along the rectangle rod, the vicinity near the edge must be the reason for the present SD formation instead of the electromigration effect.

Let us consider the SD formation dynamics through the flashing and annealing process. Si atoms at the top surface edge are expected to detach preferentially rather than those at the center part by heating, and some of the unbonded Si atoms migrate toward the center of the top surface of the rod. At last, a top surface becomes convex, and steps running parallel to [112] are formed. At the bottom surfaces, migrating adatoms were favorable to attached to the bottom corners, and the bottom surface become concave. Thus, steps running parallel to the rod, i.e., [112] direction, are expected near the corner edges of both the top and the bottom surfaces. The observation of SD near the edge indicated that monatomic steps running parallel to the rod were formed there. Since the vicinal angle corresponding to the edge deformation could not be detected by present RHEED measurements, the angle should be smaller than 1°.

In addition to the “16 × 2” SD structure, interestingly, we have observed the formation of the {35 47 7} facets at the corner edge between horizontal (110) and vertical {111} surfaces. The Si{35 47 7} surface was first reported in 1979 as one of the stable high Miller index surfaces of Si crystal [7]. Very recently, Zhachuk et al. studied the atomic structure of the Si{35 47 7} surface using STM and theory, and pointed out the similarity in the structures between the Si{35 47 7} and Si(110) “16 × 2” surfaces [21].

The facet was created by the heating procedure in the present study, indicating crystal deformation by thermal equilibration. The formation of the facet will be discussed on the basis of the surface energy. As mentioned already, {35 47 7} is 0.8° off from the [112] crystal zone [Figure 2(b)]. This mean that the nominal facet surface was formed by {35 47 7} terraces and steps perpendicular to [112] direction. Since the step density by 0.8° off is fairly low, the step energy may not dominate the facet formation.

In order for {35 47 7} facets to be formed at the corner between (110) and (111) surfaces, the surface energy of the created facet must be smaller than the energy of (110) and (111) surfaces consumed by the facet formation. Thus, the following relation must be satisfied: \( \gamma_{(35 47 7)} < \gamma_{(110)} \cos \theta + \gamma_{(111)} \sin \theta \), where \( \theta \approx 10.7° \) is the angle between {35 47 7} and (110). The surface energies for Si low index surfaces have been reported several works [22–26]. Lu et al. theoretically calculated the surface energies for Si(110) and Si(111) at high temperature to be 109.5 and 124.5 meV Å\(^{-2}\), respectively [26], where no reconstruction was considered. The condition reasonably suits the present heating conditions. Substituting these values in the above inequality, the surface energy of {35 47 7} is estimated to be <132 meV Å\(^{-2}\), i.e., <120% of \( \gamma_{(110)} \). The estimated energy reasonably agrees with the surface energy (107 meV Å\(^{-2}\)) reported for the reconstructed {35 47 7} model by Zhachuk et al. [21].

Si{35 47 7}, which may have a similarity to Si(110)“16 × 2”, should be one of key facets in 3D Si structures including Si{110} designed surfaces and will be enhanced by further heating. In the case of the micro-rods with (110) top and {111} vertical surfaces, the rectangle shape is deformed and will approach the equilibrium shape surrounded by the stable surfaces.

Figure 5: Position dependence of RHEED patterns over two rods. Patterns were measured at every 1.9 μm across the rods, and only the center part of each are arranged along with the position. SD and DD indicate the single-domain and the double-domain Si(110)“16 × 2” pattern, respectively. Observed structures are color-mapped on the schematic below (see text).
V. CONCLUSION

The rectangular micro-rods, which were three-dimensionally architectured on a Si(110) substrate by photo-lithography, were cleaned in the UHV, and their surface structure has been investigated by micro-beam electron diffraction and scanning electron microscopy. As expected for the clean (110) and {111} surfaces, “16 × 2” and 7 × 7 superstructures were confirmed for the top and bottom (110) surfaces and side {111} surfaces, respectively. Interestingly, “16 × 2” pattern showed single-domain nature near the corner of the rod surfaces. The single-domain formation is explained by the monatomic steps running parallel to the rod near the edge. In addition to the (110) and {111} surfaces, {35 47 7} facets were observed at the corner edges between (110) and {111} surfaces. The facet formation, as well as the single-domain formation, should be induced by the heating procedure. The surface energy of the facet is discussed on the basis of the shape of the micro-rod. The technique for simultaneous mapping of macroscopic surface orientation and microscopic surface structures on the 3D shaped sample can be contributed for further study of the multiple faceted surfaces and progress toward the arbitrary control of the surface structures on the 3D nano structured Si.

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