Ultrahigh Vacuum Non-Contact Atomic Force Microscope Observation of Reconstructed Si(110) Surface

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We tried to observe a reconstructed Si(110) surface using an ultrahigh-vacuum (UHV) non-contact atomic force microscope (NC-AFM). The Si(110) surface has several characteristic structures, such as the 16×2 , $(17, 15, 1) 2 \times 1$, 1×1 , zigzag structures. We succeeded in the AFM observation of the surface formed upon annealing the samples in direct current heating so as to clean the surface. We obtained the same structures in the AFM observation as the proposed 16×2 model of a Si(110) reconstruction in a STM observation. The UHV NC-AFM results demonstrate that the Si(110) surface has a characteristic 16×2 structure for the first time observation.
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1. Introduction

The scanning tunneling microscope was introduced 23 years ago,¹⁾ and many researchers have already examined a wide variety of crystalline surfaces at an atomic resolution. In the semiconductor cleaning process, almost every surface of the low-index planes is reconstituted. As a result, a superstructure is formed on the surface which is different from the atomic arrangement in the bulk crystal. The most well-known superstructure is a Si(111) 7×7 , and the DAS model has been proposed for the structure.²⁾ Several studies on Si(110) using low-energy electron diffraction (LEED), reflection high-energy electron diffraction (RHEED) and scanning tunneling microscopes (STM) have been reported. So far, a lot of researchers had to pay their efforts to get atomic images because the Si(110) plane is very sensitive to impurities. There are many theoretical atomic arrangements, and the sample preparation process is complicated. Yamamoto, *et al.* have already reported in 1980 that the Si(110) surface has a characteristic 16×2 structure, as revealed by RHEED.³⁾ The ideal Si(110) surface has dangling bonds in the top atomic layer. The top atomic layer of the surface forms a zigzag structure in the $[\bar{1}10]$ direction. A tetrahedron is constructed from unit cells; one side of the tetrahedron is 0.54 nm long in the $[001]$ direction and the other side is 0.38 nm long in the $[\bar{1}10]$ direction. A large unit cell is disposed on two symmetrical lines in the $[\bar{1}1\bar{2}]$ or the $[1\bar{1}2]$ direction. These lines make a turn-around point at an angle of 70.5 degrees.⁴⁾ Other clean Si (110) surfaces have a characteristic 1×2 ,^{5,6)} 5×4 ,⁵⁻⁸⁾ 1×5 ,^{4,7,9-12)} $(17, 15, 1) 2 \times 1$ ^{6,13-15)} or 32×2 structure.¹⁶⁾

In particular, the $(17, 15, 1) 2 \times 1$ structure is formed on the vicinal plane while the 16×2 structure is formed on the upper and lower terraces. The step edges are arranged straight along the $[\bar{1}1\bar{2}]$ or the $[1\bar{1}2]$ direction. Small terraces appear on each step edge. The terrace width is 2.5 nm and the step height is 0.19 nm. It has been reported that a phase transition occurs from the 16×2 structure to the 1×1 structure at

1043 K.¹⁷⁾ The 16×2 structure of the Si(110) surface has been investigated by LEED,¹⁸⁾ RHEED¹⁶⁾ and STM.^{3,13-15,18)} Moreover, Packard, *et al.* have proposed the 16×2 structure model for Si(110) in 1997.¹⁹⁾

Although these studies have done with LEED, RHEED or STM, nobody has used AFM for them. We are very interested in observing the reconstructed surface of the Si(110) using NC-AFM.

In case of the distance between the sample and the cantilever is farther than 1 nm, that interaction force is a Van der Waals force only. In case of the distance between the sample and the cantilever approximate to about 0.8 nm, a covalent bond force acts as the interaction forces.²⁰⁾ In using the NC-AFM with a frequency modulation detection, we can control the distance between the sample and the cantilever by adjusting frequency shifts. In case of the observation of the Si surface using the Si cantilever, the interaction forces between the surface and the cantilever depends on the distance from the cantilever to the sample.

Usually in case of the observation of a Si surface, the STM observes an electron cloud which is formed from outer electron of Si atoms; observing the surface equivalent charge density from the Si surface. On the contrary, the NC-AFM observes the physical attractive force or the chemical interaction force generated between the sample and the Si cantilever. It seems that this physical attractive force is the Van der Waals force, and the chemical interaction force is the covalent bond force, respectively.

In this study, we want to confirm the differences between NC-AFM observations and conventional STM results by adjusting frequency shifts of the Si cantilever of NC-AFM. We first obtained UHV NC-AFM images of a Si(110) surface and then elucidated the characteristic structures of Si(110), namely the $(17, 15, 1) 2 \times 1$ step, the 16×2 structure and the 32×2 structure. In order to study the superstructure of a reconstructed Si(110) surface, it is most important to clean the sample surface by annealing it at 900 K. After cleaning the Si surface, we were able to clearly observe the structural change from the meso-crystal structure to the 16×2 structure.

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Fig. 1 The block diagram of the NC-AFM system.

Fig. 2 NC-AFM image of the meso-crystal of Si (110) surface treated by process #1. Frequency shift was set at -171 Hz.

2. Experimental

In the experiment, we used a UHV NC-AFM system (JEOL, JSPM-4610A), which is schematic design shown in Fig. 1. The base pressure was less than 2×10^{-8} Pa in the AFM measurement chamber. We have done NC-AFM observation at an attractive force gradient of around 10^{-3} N/m with the scanning speed of more than 1.0 s per line. We also used a commercial naked-type conductive Si cantilever (Mikromash NSC11). The cantilever had a resonance frequency of about 280–310 kHz, and the spring constant was about 48 N/m. The AFM system was equipped with an optical deflected detector and a frequency modulation detector with a phase-locked loop (PLL) for detecting frequency shifts. High-sensitivity detection depends on the Q factor of the cantilever, and the sensitivity to the atomic force gradient increases as the Q factor increases.²¹⁾ In our system, the Q factor of the cantilever was measured to be about 3000. This value was sufficient to observe this Si surface with high detection sensitivity.

In the sample treatment, we used an n-type double-sided mirror polished Si (110) wafer (FerroTec Silicon Co.), whose electrical resistivity was 0.5–5 Ω -cm. This Si substrate doped with phosphor. A piece of wafer was cut into 10×2 mm², and the thickness of the sample was about 0.3 mm. In order to degas the samples, they were heated to a temperature of around 800 K in an UHV environment for 8 hours by direct current heating. At the first step of the heating treatment so as to clean the surface, the samples were flush-heated three times in a direct current flow at around 1500 K for 10 s (total duration: 30 s). Then, we subjected the samples to one of three processes.

In the first process, the sample was cooled very quickly from 900 K to room temperature, which calls “process #1 sample”. This process means that we stopped applying the current for the heating after sample flushing. In the second process, the sample was cooled slowly from 900 K to room temperature, which calls “process #2 sample”. The applying current for the sample decreased per 0.1 A from 0.9 to 0 A. And the decreasing span for the sample was 30 seconds per

0.1 A. In the 3rd process, the sample was annealed at a temperature of around 900 K and kept to anneal it for 3 min. Then, this sample was cooled slowly to room temperature, which calls “process #3 sample”.

3. Results and Discussion

3.1 Quick cooling after flush heating (process #1 sample)

Figure 2 shows an NC-AFM image of a Si(110) surface subjected to process #1. We took the image at a force gradient of about 51.8×10^{-3} N/m (Frequency shift: -171 Hz) and a resonance frequency of about 316 kHz. We found only the meso-crystal structures of the Si(110) surface. We could not see anything discriminative superstructures such as the 16×2 or 2×1 . The Si(110) surface often self-organizes into a 16×2 reconstruction or surface meso-crystal. The Si (110) surface cools off rapidly after flushing, the surface does not show a rearrangement structure. We could confirm only the meso-crystal structure without exception in case of the sample rapidly cooling off after flushing in Fig. 2. The meso-crystal is an intermediate state before self-organizing into a superstructure. It is necessary to give a sample annealing time for a longer time. The reason why we cannot confirm the superstructures in the surface is not enough to give the energy for the sample self-organization. It seems that we can observe 16×2 structure or 2×1 structure by a longer annealing time. In addition, in order to get a complete clean surface of the sample, we have to study the dependence of annealing time of the sample.

3.2 Slow cooling after flush heating (process #2 sample)

Figure 3(a) shows an NC-AFM image of a Si(110) surface subjected to process #2. Figs. 3(b) and (c) correspond to structure model of the Si(110) surface and a cross section profile of a white line in Fig. 3(a), respectively. We took the image at a force gradient of about 19.3×10^{-3} N/m (Frequency shift: -62 Hz) and a resonance frequency of about 310 kHz. The image shows the superstructure of the

Fig. 3 (a) NC-AFM image of the $(17, 15, 1) 2 \times 1$ step structure, 16×2 structure and meso-crystal of Si (110) surface, (b) a structure model and (c) a cross section profile of a white line in Fig(a). Frequency shift was set at -62 Hz.

$(17, 15, 1) 2 \times 1$ step structure and the 16×2 structure of the Si(110) surface. Note that the $(17, 15, 1) 2 \times 1$ structure was observed at the boundary between the vicinal terraces. In addition, the 1×1 structure and meso-crystal structures were formed in the upper and lower terraces. The step edges were arranged straight along the $[112]$ direction in one stripe or a group of stripes. Our experiment revealed that the annealing period at a temperature of around 900 K was too short to generate a complete 16×2 structure. This is because we slowly cooled the samples just after the flush heating. Also, some zigzag structures appeared on the terraces. Although high temperature-STM (HT-STM) reports also showed 16×2 , $(17, 15, 1) 2 \times 1$ and zigzag structure around 1000 K, a room temperature STM (RT-STM) has not reported yet.¹⁷⁾ We succeeded to obtain them by NC-AFM by this slow cooling after flush heating method.

3.3 Annealing at 900 K before slow cooling (process #3 sample)

Figure 4 shows an AFM image of a Si (110) surface treated by process #3. We took the image at a force gradient of about 49.2×10^{-3} N/m (Frequency shift: -144 Hz) and a resonance frequency of about 281 kHz. The characteristic 16×2 reconstruction is shown in Fig. 4. A sharp-angle corner of about 70.5 degrees is visible. Annealing for 3 min at around 900 K proved very effective for rearranging the atoms of the Si(110) surface to the characteristic 16×2 structure.

We tried to observe the Si(110) surface in detail. When we enlarged the corner of the 16×2 structure to a scanning area of $25 \text{ nm} \times 25 \text{ nm}$, we obtained the atomic structure of the corner, as shown in Fig. 5. We took the image at the same condition as that of Fig. 4. The 16×2 unit cell of the Si (110) surface are indicated by black lines in Fig. 5. The cell has two hexagonal structures, which indicated by white lines. The hexagonal lines run along $[1\bar{1}2]$ and $[11\bar{2}]$ direction. The distances between the hexagonal structures are about 1.3 nm

Fig. 4 NC-AFM image of the 16×2 structure of the Si (110) surface. Frequency shift was set at -144 Hz.

Fig. 5 NC-AFM image of the 16×2 structure of Si (110) surface. Frequency shift was set at -144 Hz.

and 5.0 nm. The acute angle formed with the 16×2 structures in the $[001]$ direction is about 70.5 degrees. The image in Fig. 5 is same as the results of the STM study.

RT-STM can observe only 16×2 and zigzag structure on Si(110) surface.¹⁹⁾ In our AFM observation of 900 K-annealing sample, it seemed that almost every structure showed only 16×2 structures. So we tried to observe another 16×2 structure in an other place. As a result, Fig. 6(a) shows the NC-AFM image of 16×2 structure. We took the image at a force gradient of about 80.6×10^{-3} N/m (Frequency shift: -236 Hz) and a resonance frequency of about 281 kHz. Secondary, we obtained the characteristic 32×2 structure, when we took the image at a force gradient of about 82.5×10^{-3} N/m (Frequency shift: -242 Hz) as shown in Fig. 6(b).

Although we tried to observe in more detail the 16×2 structure of the Si (110) surface than in the STM study, atom separation could not be achieved. We think that this structure is similar to the 16×2 structure. In the case of the 16×2

Fig. 6 (a) NC-AFM image of the 16×2 structure and (b) the 32×2 structure of the Si(110) surface. Frequency shift was set at (a) -236 Hz and (b) -242 Hz, respectively.

structure, a pentagon pair at the unoccupied state or a tetragonal pair at the occupied state is formed, and the 16×2 structures is about 5.0 nm long.⁴⁾ Usually it is easier to observe long period structures than short-period structures in case of longer distance between the tip and the sample. It was observed the 32×2 and 16×2 structure at a little difference place. The 32×2 structure predicted by an observation result of LEED, however, STM observation is not reported. In our observation, the structure we found was about 10 nm long, which seems to suggest that it was the 32×2 structure. We could observe 16×2 structure when we set a resonance frequency shift with -236 Hz, and 32×2 structure was observed when we set it with -242 Hz. As for this, it seems that the interaction force changed from the van der Waals to the covalent bond force between a cantilever and a sample in the latter case by approaching closer to a sample of a cantilever. The observation condition of 32×2 structure is about >1.0 nm closer than that of 16×2 structure in the distance between cantilever and the sample. It seems that this point is a threshold in observing 16×2 structure and 32×2 structure.

4. Conclusion

We tried to observe a reconstructed Si(110) surface using an UHV NC-AFM. We succeeded in elucidating several characteristic structures of the Si(111) surface, such as the 16×2 , $(17, 15, 1) 2 \times 1$, 32×2 , zigzag structures. We itemize the conclusions of our NC-AFM observation result.

(1) In the observation of the process of a very quick cooling

sample, we observed only the meso-crystal structures.

- (2) In the observation of the process of a slow cooling after flush heating, we observed 16×2 , $(17, 15, 1) 2 \times 1$, 1×1 , meso-crystal and zigzag structures at same time.
- (3) The observation of the process of an annealing at 900 K before slow cooling, we observed only 16×2 structures. This point is not same as other reported STM observations. We thought 16×2 structures appeared because zigzag structure grows up in the heating time.
- (4) An other area of the observation of the process of an annealing at 900 K before slow cooling, we observed both the 16×2 and the 32×2 structure. In addition, we succeed in the observation of 32×2 structure by UHV-NC-AFM for the first time.
- (5) The case of detecting force of observation is the Van der Waals, NC-AFM observation results are almost same as the reported STM observations. It seems that we can observe almost same result as reported STM observations in setting at a force of the Van der Waals detecting region. On the contrary, it seems that we can observe the 32×2 structure in setting a force of the covalent bond force detecting region.

We were able to reconfirm the proposed model of the reconstructed Si(110) structure. This is the first observation of the characteristic structure of the Si (110) surface using an UHV NC-AFM.

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