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Surface Science 493 (2001) 148–156

SURFACE SCIENCE

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A series of Ca-induced reconstructions on Si(1 1 1) surface

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Received 21 September 2000; accepted for publication 9 January 2001

Abstract

We have studied surface reconstructions induced by submonolayer Ca adsorption on a Si(1 1 1) surface using reflection-high-energy electron diffraction (RHEED) and scanning tunneling microscopy (STM). With increase of Ca coverage at substrate temperatures above 550°C, the RHEED pattern changed successively: clean $7 \times 7 \rightarrow 3 \times 1 \rightarrow 5 \times 1 \rightarrow 7 \times 1 \rightarrow 2 \times 1$. The 3×1 pattern transformed into a $3 \times$ "2" pattern including half-order streaks by cooling down to room temperature. The STM images of these reconstructions commonly showed striped structures with the periodicities corresponding to the RHEED patterns. Each stripe in the $3 \times$ "2", 5×1, 7×1, and 2×1 structures is composed of several rows. The twofold periodicity along the stripes of the $3 \times$ "2" phase was observed in filled-state images as well as in empty-state ones; simple protrusions are arranged with the double periodicity in the empty-state image, while a dimerization of small protrusions occurs along the stripes in the filled-state image. From these results, atomic structure models for these Ca-induced reconstructions are suggested.

Keywords: Alkaline earth metals; Reflection high-energy electron diffraction (RHEED); Scanning tunneling microscopy; Silicon; Surface relaxation and reconstruction

1. Introduction

It has been known that divalent metal (DM) atoms induce several one-dimensional reconstructions ($n \times 1$ superstructures) on a Si(1 1 1) surface [1–6]. In particular, a 3×1 reconstruction was found to occur commonly by adsorption of Yb [1], Sm [2], Mg [3], Ca [4,6], Sr [4], and Ba [5] as well as alkali metals (AM) [7,8]. Yb- and Sm-induced 3×1 reconstructions showed half-order streaks in low-energy electron diffraction (LEED) patterns [1,2]. Although the 3×1 reconstructions induced by other DM showed no half-order streaks in LEED, studies with reflection-high-energy electron diffraction (RHEED) [4] and spot-profile-analyzing LEED [6] revealed that such streaks are seen also for the 3×1-Ca and -Sr phases. The double-periodicity modulation along stripes that corresponds to the half-order streaks was observed in

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0039-6028/01/$ - see front matter
PII: S0039-6028(01)01203-1

the empty-state scanning tunneling microscopy (STM) images of the 3×1-Mg [9] and 3×1-Ca [6]. So these 3×1 phases induced by DM should be denoted as $3 \times$"2"-DM. Furthermore, the $3 \times$"2"-Mg structure is considered to have an atomic arrangement similar to those in the 3×1 structures induced by AM atoms or Ag atoms, because the LEED I–V curves of the $3 \times$"2"-Mg phase were reported to be quite similar to those from the 3×1-Li, -Na, and -Ag phases [3]. Therefore, it may be considered that these $3 \times$"2"-DM structures have common atomic arrangements which are similar to the 3×1-AM ones, and that divalence of adsorbates induces the double-periodicity modulation along the stripes of the 3×1 structure, resulting in the $3 \times$"2" structure at room temperature (RT).

Other than the 3×1 or $3 \times$"2" structures, different reconstructions appear depending on the species of adsorbates. In particular, a $\sqrt{3} \times \sqrt{3}$ structure was reported for Sm adsorption. This is due to the variation of valence characteristic for rare-earth metal atoms. Indeed, it has been revealed that Sm is almost divalent in the $n \times 1$ structure, but completely trivalent in the $\sqrt{3} \times \sqrt{3}$ structure [2]. Thus it is suggested that divalence of the adsorbed metal atoms play a crucial role in forming and stabilizing these one-dimensional $n \times 1$ structures.

Various structure models have been proposed for the Si(1 1 1)–3×1-AM structures. Recently, plausible models were restricted to the following two, double-π-bonded chain (DπC) model and honeycomb chain-channel (HCC) model [10], mainly because the Si atom densities in the 3×1-(AM, Ag, Ca, or Mg) phases were reported to be 4/3 ML from the STM images [10]. The latter model was examined about its surface electronic bands, charge distribution, and simulated STM images by the first-principles calculations, which agreed well with the experimental results [11,12]. In particular, STM images were well reproduced including the correspondence between the filled-state and empty-state images; the apparent dark channels between the protrusion rows are observed at the identical positions in both images [13]. On the other hand, such a first-principles calculations were not performed for the DπC model, although the filled-state STM images of the 3×1-AM were explained in terms of this model [6]. Its interpretation, however, seems to be inconsistent with the experimental results in a way that the apparent dark channels in the filled-state image and empty-state images may not be observed at the identical positions. Therefore the most plausible model for the 3×1 structure is the HCC model at the present.

In this paper, we report on the results for a series of Ca-induced reconstructions on a Si(1 1 1) surface using RHEED and STM. RHEED observations have revealed that 3×1, 5×1, 7×1, and 2×1 reconstructions were formed with increase of Ca coverage on a Si(1 1 1) substrate at elevated temperatures. STM observations have also revealed that these reconstructions have striped structures commonly, and each stripe is composed of several rows. In particular, the 3×1-Ca phase has been found to transform into the $3 \times$ "2"-Ca phase by cooling down to RT, and a double-periodicity modulation along the stripes has been observed in filled-state as well as empty-state STM images. The filled-state images shows similarities to those for the 3×1-AM phases. From these results, we suggest structure models of these striped reconstructions.

2. Experimental

Experiments were carried out in an ultrahigh vacuum (UHV) chamber with a base pressure lower than 5×10^{-11} Torr equipped with RHEED and STM (UNISOKU USM-802 type) systems. In this chamber, RHEED and STM measurements could be performed in situ on a single stage, at temperatures ranging from RT to 800°C, and during deposition of metals. The substrates used were highly doped n-type Si(1 1 1) wafers with a miscut angle less than 0.5°. The surface was cleaned by flash heating up to 1260°C after sufficient outgassing below 500°C. After these treatments, RHEED patterns and STM images showed a well-ordered 7×7 superstructure. The azimuth of the substrate was determined from the difference between faulted and unfaulted halves of the 7×7 clean surface in the filled-state STM images.

Calcium was evaporated from an alumina-coated W basket. Ca coverage Θ_{Ca} was calibrated by the duration time in deposition at 600°C on an assumption that Θ_{Ca} for completing the 3×1-Ca phase was the same as the metal coverage (1/3 ML) for other 3×1 phases induced by Na [14], K [15], Ag [16], and Mg [3,9]. This value also agrees with Ca coverage for a 3×1 reconstruction formed by desorption of F from CaF_2-adsorbed Si(1 1 1) surface [17]. Tungsten tips used for STM were etched electrochemically in KOH solution and cleaned by heating in UHV.

3. Results

3.1. Reflection-high-energy electron diffraction observations

Structural transformations on the Si(1 1 1) surface induced by Ca adsorption observed in the present study are summarized in Fig. 1. This formation phase diagram is based on the RHEED observations during Ca deposition onto the clean Si(1 1 1)-7×7 surface held at various constant temperatures. Between RT and 550°C, no additional spots of superstructures appeared; the 7×7 superspots just gradually weakened with Ca deposition, then the pattern changed into a so-called δ-7×7 structure, and finally into a 1×1 [4]. On the other hand, Ca deposition above 550°C caused drastic changes in RHEED pattern: 7×7 (clean) → triple-domain(denoted as TD hereafter)-3×1 TD-5×1 → TD-7×1 → TD-2×1. When Ca-adsorbed Si(1 1 1) surface was annealed above 600°C for a prolonged time, the superstructures changed in the opposite order due to Ca desorption from the surface [4]. This behavior is similar to that for CaF_2-adsorbed surfaces [17], but different from that in the previous LEED observations of the Ca-adsorbed surfaces [6], where the 7×1 phase appeared between the 3×1 and 5×1 phases.

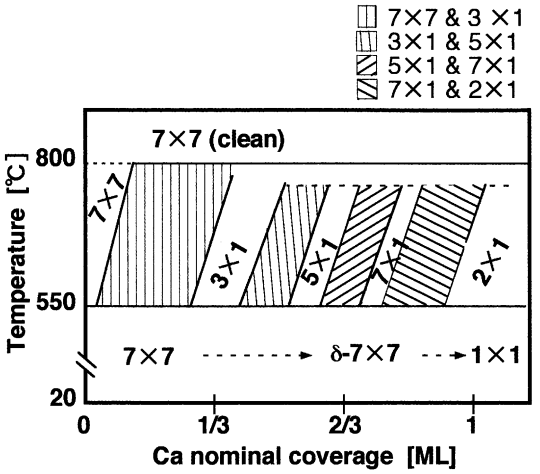


Fig. 1. Formation phase diagram of Ca/Si(1 1 1) system during isothermal adsorption. Hatched are the regions where the neighboring phases coexist.

Fig. 2 shows RHEED patterns of these structures induced by Ca deposition at 700°C; the photos were taken at RT. While Fig. 2a contains half-order streaks (indicated by arrowheads) as well as 3×1-superspots (i.e., $3 \times$ "2") at RT, only the streaks reversibly disappeared above about 300°C, remaining a 3×1 pattern. Therefore a temperature-induced reversible transition ($3 \times 1 \rightleftarrows 3\times$ "2") is found to occur. Such half-order streaks in electron diffraction patterns have been observed also for the Si(1 1 1)–$5 \times$ "2"-Au [18] and Si(1 1 1)–$8 \times$ "2"-In superstructures [19], both of which are known to be highly anisotropic in atomic/electronic structures. The origin of these streaks will be discussed with STM results later. Here we denote these superstructures generally as $n \times 1$ ($n = 3, 5, 7, 2$), including the $3 \times$ "2" phase for brevity. In particular, as for the 2×1 phase, it cannot be distinguished from a 2×2 structure only by the RHEED patterns shown in Fig. 2d. However, the notation of TD-2×1 is appropriate as described later in the STM measurements.

As shown in Fig. 1, the neighboring phases coexisted on the surface at intermediate Ca coverages where the fractional RHEED spots of the two phases, e.g., the 5×1 and 7×1, were connected each other by tails along the $\langle 1\,1\,\bar{2} \rangle$ directions [4]. This means that phase separations between the neighboring phases do not occur, rather the two phases are mixed in microscopic way at the intermediate coverages. This will be seen in STM images later where the stripes of fivefold and sevenfold periodicities are mixed microscopically. Only between the 7×7 and 3×1 phases, the fractional

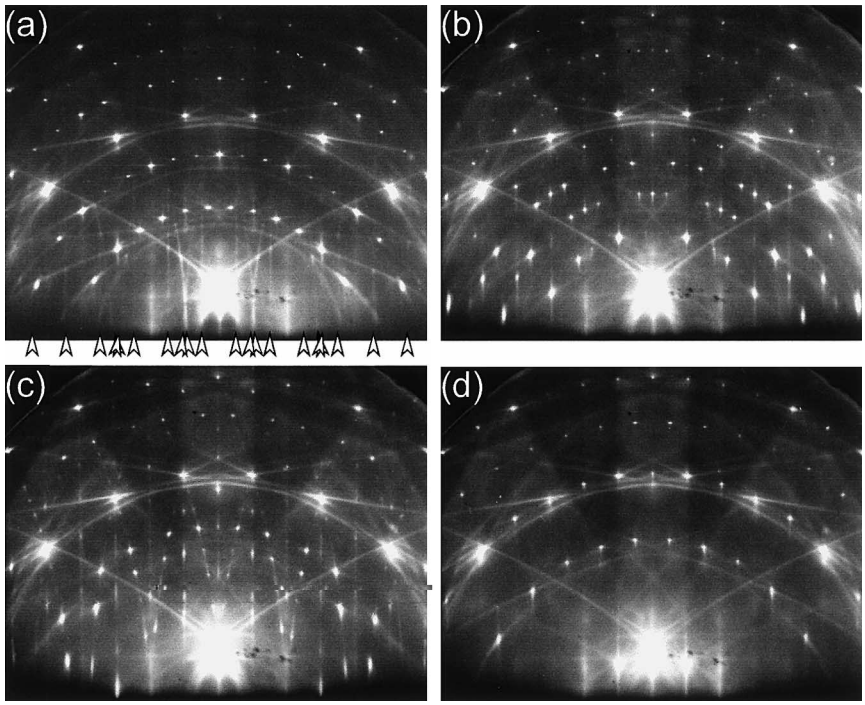


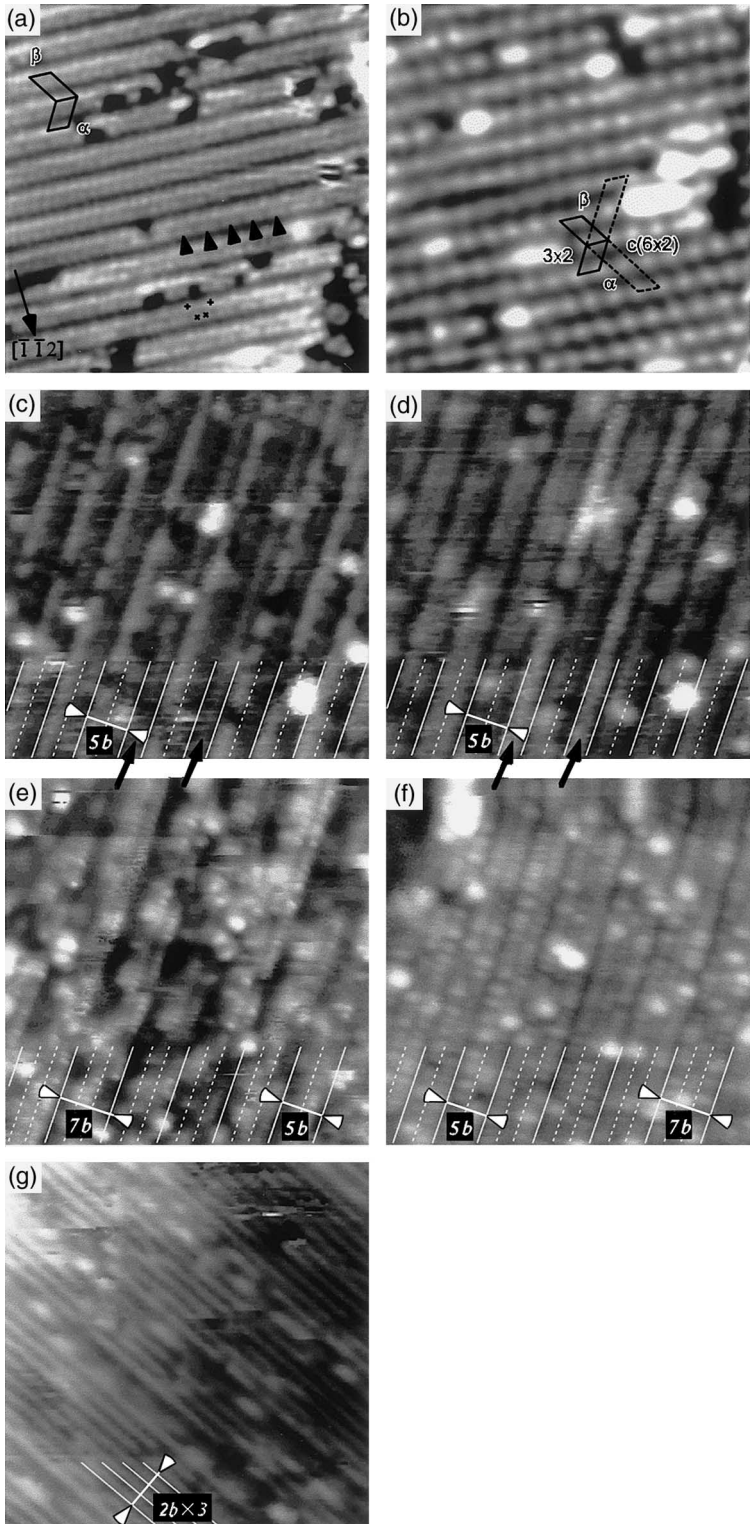
Fig. 2. RHEED patterns of the (a) $3 \times$ "2"-Ca, (b) 5×1-Ca, (c) 7×1-Ca, and (d) 2×1-Ca surfaces, observed at RT with electron energy of 14.5 keV and incident azimuth of $\langle 1\,1\,2 \rangle$. The arrowheads in (a) indicate half-order streaks.

RHEED spots coexisted separately, which means a phase separation between the two phases.

3.2. Scanning tunneling microscopy observations

Fig. 3 shows STM images of these Si(1 1 1)–$n \times 1$-Ca structures observed at RT. Each image consists of stripes with a separation of nb, corresponding to the RHEED patterns, where $b = a \sin 120° = 3.33$ Å and $a = 3.84$ Å, a being the length of primitive vectors on an ideal Si(1 1 1)-1×1 surface. In particular, on the $3 \times$ "2" surface, the double-periodicity $2a$ modulation along the stripes is clearly seen in the empty-state images (Fig. 3b). The phases in this double periodicity between the neighboring stripes are scarcely correlated. This is why the half-order streaks appear, instead of half-order spots, in RHEED patterns. In other words, there exist two types of 3×2 stripes (α and β as shown in Fig. 3b), which are arranged randomly. In the empty-state image, only one protrusion is observed in the 3×2 unit cell. This is consistent with the previous result of Ca-adsorbed surfaces [6], and similar to that in the empty-state STM images of a $3 \times$ "2" surface induced by Mg [9]. Each stripe in the filled-state image (Fig. 3a) consists of doubled rows similar to the zigzag chains in the AM-induced 3×1 structures on Si(1 1 1) [13,20,21]. However, the two rows constructing this zigzag chain are inequivalent in the way of producing the double periodicity, while the two rows in the 3×1-AM phases are equivalent without such a modulation along the rows [13,20,21]. In more detail, protrusions in the brighter one of the two rows in Fig. 3a appear to be arranged with a spacing of $1a$, but protrusions in the darker rows appear to be dimerized along the rows as indicated by black arrowheads. In this way, the $2a$ modulation looks different from each other in the empty- and filled-state images.

We observed no $3 \times$ "2" domains in twin relation with respect to a $(1\,1\,\bar{2})$ plane (parallel to the


(a)
β
α
[1̄ 1̄ 2]
(b)
β
3x2
c(6x2)
α
(c)
5b
(d)
5b
(e)
7b
5b
(f)
5b
7b
(g)
2b×3

stripes). In other words, the $3\times$"2"-Ca structure has a definite orientation with respect to the substrate so that a brighter row, a darker row, and a darkest channel are arranged in the order in $\langle\bar{1}\,\bar{1}\,2\rangle$ directions, as shown in Fig. 3a, but not in the opposite $\langle 1\,1\,\bar{2}\rangle$ directions. This is caused by an influence of the substrate that has a threefold (not sixfold) rotational symmetry. In addition, from the STM images of an area where the $3\times$"2"-Ca and 7×7 domains coexisted, it was also revealed that the darkest channels in the filled-state images were observed at the identical positions in the empty-state ones. This correspondence is the same as those for the 3×1-Li and 3×1-Ag structures [13]. However, the channels are not valleys in morphology, but due to electronic effect, which is explained using the HCC model for the 3×1-AM structures [11,12].

The filled-state (Fig. 3c) and the empty-state (Fig. 3d) STM images were observed at almost the same area for the 5×1-Ca surface. In both images, each stripe of 5*b* width is composed of two rows with almost the same width. However, these two rows look inequivalent in height in the filled states while they appear to be equivalent in the empty states. As in the case of the $3\times$"2" surface mentioned above, the dark channels between the stripes were observed at the same positions in both of the empty- and filled-state images. There are two positions having an irregular periodicity as indicated by black arrows in Fig. 3c, which is a kind of out-of-phase boundary.

While Figs. 3e and f correspond to the surface exhibiting the 7×1-RHEED pattern (Fig. 2c), they contain the 5×1 stripes partially as well as the 7×1 domains, as indicated in the figures. In the 7×1 domains in both images, the stripes of 7*b* wide are composed of three rows with almost the same width. Similar to the 5×1 phase, these rows appear to be inequivalent in the filled-state image (Fig. 3e) but equivalent in the empty-state image (Fig. 3f).

The images of the 2×1-Ca structure were obtained only in the filled states. The STM image (Fig. 3g) corresponding to Fig. 2d shows that this structure consists of stripes separated by 2*b* although no corrugations were observed along the stripes. Then it may be appropriate that this surface is denoted as a 2×1, not a 2×2, at variance with the previous report [6].

4. Discussion

The STM images of the $3\times$"2"-DM structures have been observed by some groups (Yb [1], Sm [2], Mg [9], and Ca [6]). All these images are similar to our empty-state images of the $3\times$"2"-Ca presented here (Fig. 3b). They consist of stripes of 3*b* wide, which have a modulation with 2*a* periodicity along the stripes. In contrast, the 3×1-AM structures do not show such corrugations along the 3*b*-stripes in empty states [13]. The filled-state STM images of the $3\times$"2"-DM structures have not been observed so far. Since the filled-state image Fig. 3a observed for the first time show finer structures, they provide additional information for structure determination. First, the stripes showing a zigzag-chain structure are separated by dark channels. This looks similar to those for 3×1-AM structures [13]. Second, two rows constructing each zig-zag chain are inequivalent in images, while these are equivalent in 3×1-AM structures [13]. One of the two rows has a 2*a* modulation by a kind of dimerization of small protrusions. These features enable us to distinguish the $3\times$"2" from a 3×1 structure in filled-state images. Third, these two rows and dark channels have a definite arrangement determined by threefold (not sixfold) rotational symmetry of the substrate. Finally, apparent dark channels are observed at the identical positions in the dual-polarity images, which is the same as those for the 3×1-AM structures [13]. These similarities in the filled-state STM images

Fig. 3. STM images of the (a,b) $3\times$ "2"-Ca (11×11 nm^2), (c,d) 5×1-Ca (16.5×16.5 nm^2), (e,f) 7×1-Ca (16.5×16.5 nm^2), and (g) 2×1-Ca surfaces (16.5×16.5 nm^2), observed at RT. Tip-bias voltages V_T and tunneling currents I_t are as follows: (a) +1.80 V and 0.25 nA, (b) −1.80 V and 0.20 nA, (c) +1.50 V and 0.15 nA, (d) −1.50 V and 0.22 nA, (e) +2.00 V and 0.15 nA, (f) −2.00 V and 0.16 nA, and (g) +1.80 V and 0.20 nA.

between 3×1-AM and $3 \times$ "2"-Ca structures support an idea that these structures are very similar to each other in atomic arrangements. The difference between the 3×1-AM and $3 \times$ "2"-DM is only the double-periodicity modulation along the stripes. It should be noted that divalence of adsorbed metal atoms might cause a $2a$ modulation along the rows due to instability of one-dimensional metals (Peierls-like instability). The 3×1-DM should be metallic due to electron counting consideration, while the $3 \times$ "2"-DM can be semiconducting, which will be reported elsewhere together with our photoemission spectroscopy data.

From the information described above, we propose a structure model for the $3 \times$ "2"-Ca reconstruction, as shown in Fig. 4a, based on the HCC model proposed for the 3×1-AM surfaces [11]. Compared to the original HCC model for the 3×1-AM reconstructions, the arrangement of top-layer Si atoms is the same, but the Ca-adsorbed sites are slightly different in the following points: Two Ca atoms (M_A and M_B) per 3×2 unit cell (dashed parallelograms) are adsorbed at inequivalent sites, slightly shifted from T_4 sites toward the opposite directions perpendicular to the stripes, i.e., $[1\,1\,\bar{2}]$ and $[\bar{1}\,\bar{1}\,2]$ directions. One (M_A) of the two Ca is raised relatively. This model can explain well the STM images of the $3 \times$ "2"-Ca surface reported here, in the same manner as the HCC model reproducing STM images of the 3×1-AM surfaces. In Fig. 4a, the protrusions in the filled-state STM images are indicated by dashed circles. The four protrusions (labeled as A1, A2, B1, and B2) per a 3×2-unit cell correspond to the dangling-bond states of the Si atoms

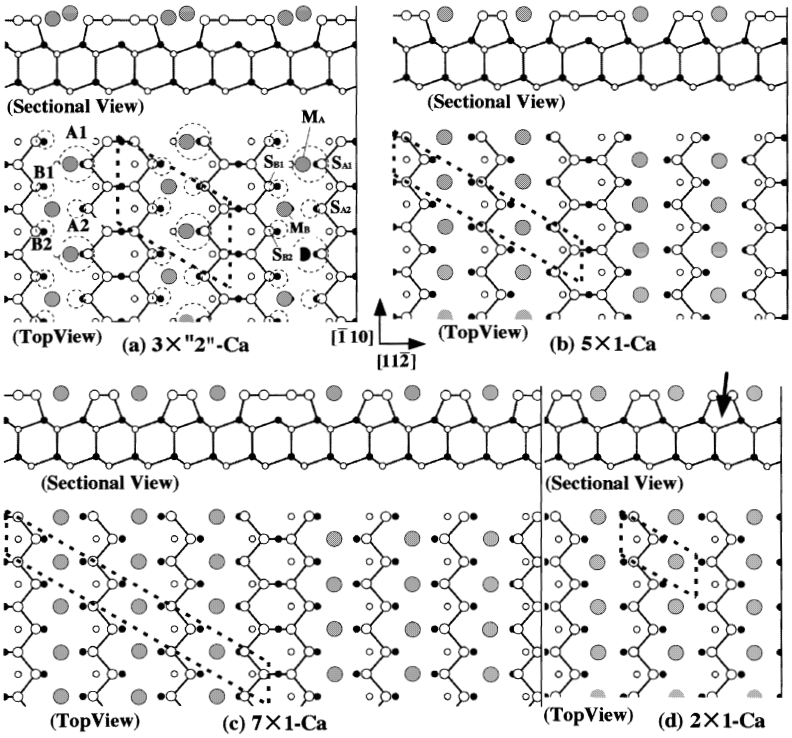


Fig. 4. Proposed structure models for the Si(1 1 1)–$n \times 1$-Ca surfaces. (a) A model for the $3 \times$ "2"-Ca surface, modified from the HCC one, (b) the 5×1-Ca, (c) the 7×1-Ca, and (d) the 2×1-Ca surfaces. Dashed lines indicate a unit cell of the corresponding structures. Gray and other circles represent Ca and Si atoms, respectively, and some of which are labeled in (a) as M_A, S_{B2}, etc. Dashed circles labeled as A1, B2, etc. in (a) indicate protrusions in the filled-state images.

(S_{A1}, S_{A2}, S_{B1}, and S_{B2}) constructing the honeycomb chains, like in the HCC model for the 3×1-AM structures. However, shift of adsorbed Ca causes slight modifications in images: the brightest one (A1) of these protrusions corresponds to the Si (S_{A1}) bonding to Ca (M_A) shifted toward both [1 1 $\bar{2}$] and [1 1 1] directions. This raised Ca atom can enhance the apparent height of this protrusion in STM images. The other Ca (M_B) approaches to the two Si atoms (S_{B1} and S_{B2}), so that the protrusions (B1 and B2) appear to be dimerized. The smaller protrusion A2 remains almost unchanged. In addition, the definite orientation of the structure with respect to the substrate can be explained in this model by preferential adsorption of Ca on T_4 rather than H_3 site. If Ca atoms were positioned close to H_3 sites, the orientation of the filled-state images would be reversed. On the other hand, one protrusion per 3×2 unit cell in the empty-state images (Fig. 3b) is considered to correspond to the raised Ca atom (M_A), because it is similar to the 3×1-AM case where the bright rows in the empty-state image of the 3×1-AM correspond to AM ions in the HCC model. Similarly, the identical positions of the dark channels observed in both of the filled- and empty-state images can be explained by this model.

Because the 5×1-Ca and 7×1-Ca structures in both of the filled- and empty-state images consist of double and triple rows, respectively, it is suggested that the fivefold (5×1) and sevenfold (7×1) stripes are formed by 1:1 and 1:2 arrangements, respectively, of threefold (3×1) and twofold (2×1) stripes. Because these (3×1) and (2×1) stripes look similar in apparent height, the top Si layer in the 2×1 structure would have a height similar to that of the honeycomb chain in the $3 \times$ "2" structure in Fig. 4a. Thus we suggest a model for the 2×1-Ca phase as shown in Fig. 4d, which has five-membered Si rings as indicated by an arrow in the sectional view. This Si chain is called a Seiwatz chain [22], which is a unit of the Seiwatz model of the 3×1-AM structure although it was discarded from the 3×1-AM structure model due to the difference in the top Si atom density. This model has a density of the top-layer Si atoms of 1 ML. Ca atoms are assumed to sit on T_4 sites as in the other $n \times 1$-Ca structures. This results in the Ca coverage being 1/2 ML. It should be noted here, however, that our RHEED experiments indicated the Ca coverage for the 2×1-Ca phase to be around 1 ML (see Fig. 1). This discrepancy might be due to a smaller sticking coefficient of Ca onto the elevated-temperature substrate, especially at higher coverage ranges. Actually, Saranin et al. reported that the 2×1-Ca structure was formed at Ca coverage below 0.6 ML [6].

Based on this 2×1-Ca structure model (Fig. 4d) combined with the $3 \times$ "2" model (Fig. 4a), we propose models for the 5×1-Ca structure (Fig. 4b) and the 7×1-Ca structure (Fig. 4c). The common feature of these models is that Si chains are separated by Ca atomic rows. These Si chains are honeycomb chains and Seiwatz chains, which are components of the structure models proposed for the $3 \times$ "2"-Ca (Fig. 4a) and the 2×1-Ca (Fig. 4d), respectively. Based on these models, our STM images can be explained by the idea similar to that used for the $3 \times$ "2"-Ca: bright rows in the empty-state images correspond to Ca ions, like AM ions in the 3×1-AM structures, and those in the filled-state images correspond to the dangling-bond states of Si atoms in the honeycomb and Seiwatz chains. This is consistent also with the equivalent apparent heights of the two and three rows in the 5×1 and 7×1 stripes in the empty-state images, but seem to be inconsistent with the inequivalence of these rows in the filled-state images. The latter could be explained by deformation of Si chains, such as buckling of Seiwatz chains [22]. In the Seiwatz model, the 3×1-AM structures are stabilized by this deformation [22]. These models should be confirmed by diffraction experiments.

5. Conclusion

We have studied a series of Ca-induced reconstructions on Si(1 1 1) surface using RHEED and STM. It has been found that 3×1, 5×1, 7×1, and 2×1 structures are formed in the order with increase of Ca coverage at elevated substrate temperatures. The 3×1 structure transforms into a $3 \times$ "2" below about 300°C. From the STM images, these structures are found to be striped

structures that are composed of several rows. In particular, the $3\times$"2"-Ca structure appears to be the same as $3\times$"2"-Mg and slightly modified from the 3×1-AM structures. We have proposed atomic models for these structures.

Acknowledgements

This work has been supported in part by Grants-In-Aid from the Ministry of Education, Science, Culture, and Sports of Japan, especially through the Creative Basic Research Program (no. 08NP1201) conducted by Professor Katsumichi Yagi of Tokyo Institute of Technology. We have been supported also by CREST (Core Research for Evolutional Science and Technology) of the Japan Science and Technology Corporation (JST) conducted by Professor Masakazu Aono of Osaka University and RIKEN, and by the Special Researcher of Basic Science Program in RIKEN.

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