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Structure and peculiarities of the $(8 \times n)$-type Si(001) surface prepared in a molecular beam epitaxy chamber: a scanning tunnelling microscopy study

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A clean Si(001) surface thermally purified in the ultra-high-vacuum molecular beam epitaxy chamber has been investigated by means of the scanning tunnelling microscopy. Morphological peculiarities of the Si(001) surface have been explored in detail. A classification of surface structure elements has been carried out, the dimensions of the elements have been measured, and relative heights of the surface relief have been determined. A reconstruction of the Si(001) surface prepared in the molecular beam epitaxy chamber has been found to be $(8 \times n)$. A model of the Si(001) – $(8 \times n)$ surface structure is proposed.

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Investigations of clean silicon surfaces prepared in conditions of actual technological chambers are of great interest due to the industrial requirements to operate on nanometer and subnanometer scale when designing future nanoelectronic devices [1]. In the nearest future, the sizes of structural elements of such devices will be close to the dimensions of structure features of Si(001) surface, at least of its high-order reconstructions such as $c(8 \times 8)$. Most of researches of the Si(001) surface have so far been carried out in specially refined conditions which allowed one to study the most common types of the surface reconstructions such as (2×1), $c(4 \times 4)$, $c(4 \times 2)$ or $c(8 \times 8)$ [2 – 13]. The ambient in technological vessels such as molecular beam epitaxy (MBE) chambers is usually not so pure as in specially refined ones designed for surface studies. There are many sources of surface contaminants in the process chambers including materials of heaters and evaporators as well as foreign substances used for epitaxy and doping.

In the present paper we report the results of investigation of the Si(001) surface treated following a standard procedure of Si wafer preparation for the MBE growth.

The experiments were made using an integrated ultra-high-vacuum (UHV) system based on the Riber EVA 32 molecular beam epitaxy chamber coupled through a transfer line with the GPI 300 scanning tunnelling microscope (STM). This instrument enables the STM study of samples at any stage of Si surface cleaning and MBE growth. The samples can be moved in the STM chamber and back in the MBE chamber never leaving the UHV ambient.

The samples were $8 \times 8\,\mathrm{mm}^2$ squares cut from B-doped CZ Si(100) wafers (p-type, $\rho = 12\ \Omega\,\mathrm{cm}$). After washing and chemical treatment following a standard procedure,[1] the samples mounted on the Mo STM holder and clamped with the Ta fasteners were loaded into the airlock and transferred to the preliminary annealing chamber where outgassed at $\sim 565\,^\circ\mathrm{C}$ and $\sim 5 \cdot 10^{-9}$ Torr for about 24 hours. After that the samples were moved for final treatment into the MBE chamber evacuated down to $\sim 10^{-11}$ Torr. There were two stages of annealing in the process of sample heating – at $\sim 600\,^\circ\mathrm{C}$ for $\sim 5\,\mathrm{min}$ and at $\sim 800\,^\circ\mathrm{C}$ for ~ 3 min. The final annealing at $\sim 900\,^\circ\mathrm{C}$ was carried out for ~ 2.5 min with maximum temperature $\sim 920\,^\circ\mathrm{C}$ (~ 1.5 min). Then the temperature was rapidly lowered to $\sim 750\,^\circ\mathrm{C}$. The rate of the further cooling down was $\sim 0.4\,^\circ\mathrm{C/s}$. The pressure in the MBE chamber enhanced to $\sim 2 \cdot 10^{-9}$ Torr during the process.

The samples were heated by Ta radiators from the rear side in both chambers. The temperature was monitored with chromel-allimel and tungsten-rhenium thermocouples in the preliminary annealing and MBE chambers, respectively. The thermocouples were mounted near the rear side of the samples and *in situ* graduated against the IMPAC IS 12-Si pyrometer which measured the Si sample temperature through chamber windows.

After cooling, the samples were moved into the STM chamber in which the pressure did not exceed 10^{-10} Torr. The STM tip was *ex situ* made of the W

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wire and cleaned by ion bombardment [14] in a special UHV chamber connected to the STM chamber.

The images were obtained in the constant tunnelling current mode at room temperature. The STM tip was zero-biased while the sample was positively or negatively biased for empty or filled states mapping.

Fig.1 demonstrates the images of the surface prepared according to the above procedure. Steps with a

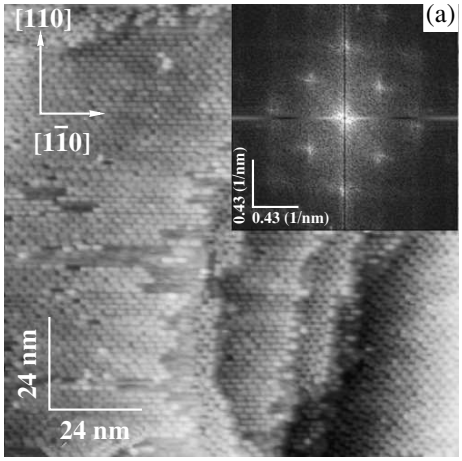

Fig.1. An STM image of the Si(001) surface after annealing (+1.9 V, 70 pA), rows run along [110] or [1$\bar{1}$0] directions; the insert shows its 2-D Fourier transform pattern

height of $\sim$ 1.4 Å are seen in the pictures. The surface is composed of rows running along [110] and [1$\bar{1}$0] directions. The rows lying on each terrace are parallel to the rows which form the terraces separated by an even number of steps and perpendicularly to the rows forming the terraces separated by an odd number of steps. Each row consists of rectangular blocks which may be considered as structural units. Reflexes of the Fourier transform of the picture (see the insert in Fig.1) correspond to the distances $\sim$ 31 Å and $\sim$ 15 Å in both [110] and [1$\bar{1}$0] directions. So the structure revealed in the long shot seems to have a periodicity of $\sim$ 31 Å that corresponds to 8 translations a on the surface lattice of Si(001) (a = 3.83 Å is a unit translation length). It resembles the Si(001)$-c(8\times 8)$ surface [5]. Reflexes corresponding to the distance of $\sim$ 15 Å ($4a$) arise due to the periodicity along the rows.

STM images obtained at higher magnifications give an evidence that the surface appears to be disordered, though.

Fig.2 shows the magnified images of the investigated surface. The rows of the blocks are seen to be situated at varying distances from one another (hereinafter, the distances are measured between corresponding maxima of features). A unit (8 × 8) cell is marked with a

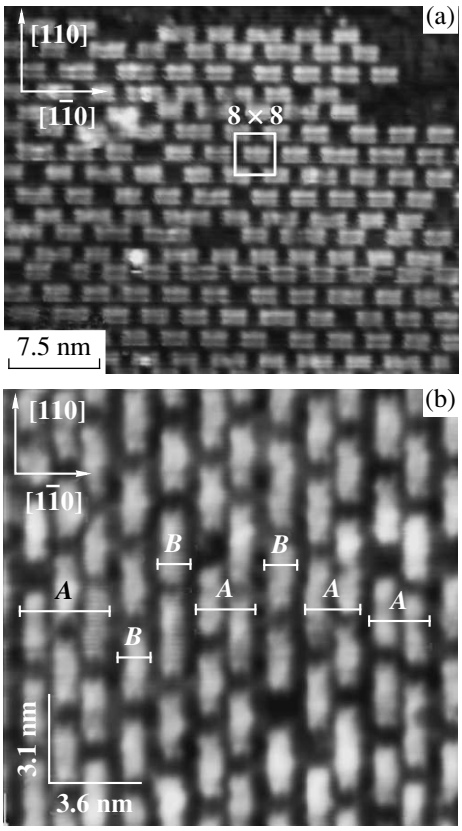

Fig.2. Empty state images of the Si(001) surface. (a) +2.0 V, 200 pA; (b) +2.0 V, 150 pA. A $c(8\times 8)$ unit cell is marked by a white box in (a); (8 × 6) and (8 × 8) structures are marked as A and B in (b)

square box in Fig.2a. The distances between the adjacent rows of the rectangles are $4a$ in such structures. Fig.2b demonstrates a surface area which contains a different structure of the rows. The adjacent rows designated as A are $3a$ apart. The rows marked as B go at $4a$ apart. Hence it may be concluded that the order and some periodicity take place only along the rows. The structure is disordered across the rows.

Fig.3 demonstrates the empty and filled state images of the same surface. Each block consists of two maxima clearly seen in both images. Extreme positions in both images are well fitted.

Before considering a model of the observed surface structure, let us dwell upon a close-up of the surface in more detail and especially on the blocks as its unit elements.

Looking at the above images one can see two types of blocks forming the surface structure – $\sim$ 15 Å ($4a$) and $\sim$ 23 Å ($6a$) long. The distance between equivalent positions of the adjacent short blocks in the rows is $8a$. If the long block appears in a row, a vacancy is formed

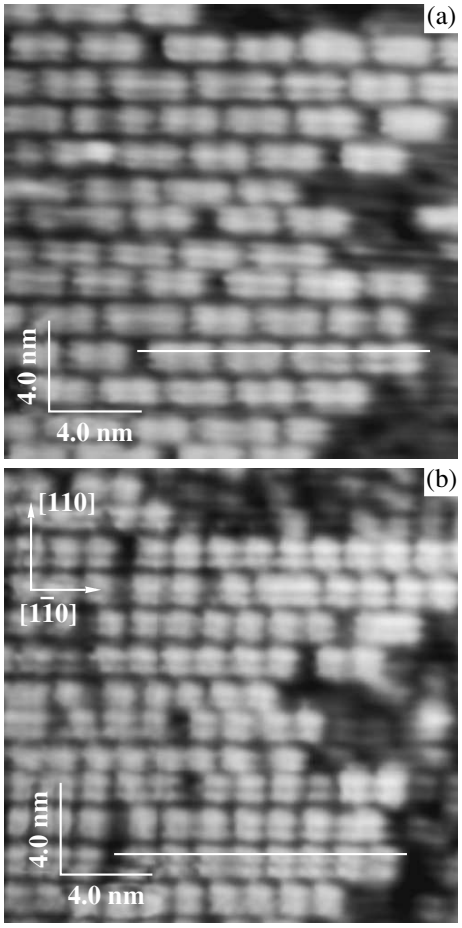

Fig.3. Empty and filled state images of the same region on Si(001) surface. (a) Empty states (+1.7 V, 100 pA); (b) Filled states (−2.0 V, 100 pA)

in the adjacent row. With this, a Fourier transform pattern of the overview image (Fig.1) looks like that for the undisturbed $c(8 \times 8)$ structure, so the mentioned irregularities likely cannot be detected by means of integral techniques, e.g. by LEED. In average, the structure resembles $c(8 \times 8)$ one.

It was found then that the rectangles are elevated over the surface through a height of at least 1.4 Å that equals the height of a monoatomic step on the Si(001) surface. The measurements made on a number of the STM images enabled the determination of all possible differences in height – within the blocks (both short or long), in gaps between adjacent blocks in a row and between neighboring rows, etc. So a surface relief was determined with an accuracy provided by the STM.

In addition, the long blocks were found to have one more peculiarity. They have extra maxima in their central regions. The maxima are not so pronounced as the main ones but nevertheless they are quite recognizable in the pictures (see Fig.2).

Fig.3b gives an evidence that each rectangle consists of two rows separated by the distance close to a. Corresponding features are seen in Fig.4 aswell and marked

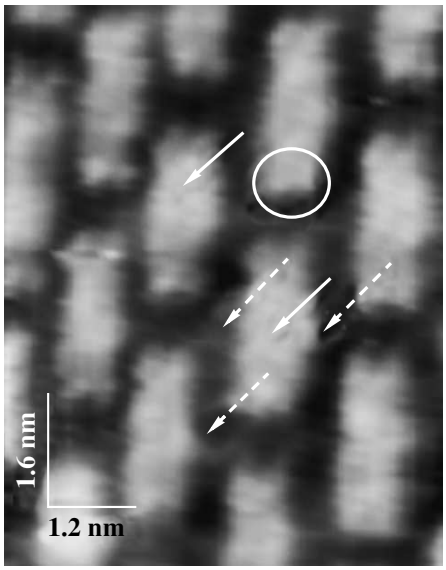

Fig.4. An empty state image of the rows (+2.0 V, 150 pA)

with a circle and solid arrows. Precise measurement of this distance is difficult because an STM image reflects the electron density distribution of atoms situated both in the outermost layer and in the layer stretching under it [15]. A total signal comprises a superposition of signals from several close atoms that sometimes results in line widening or displacement in the STM images.

And at last, an STM signal is registered in the gaps between the neighboring rows (Fig.4, indicated with the dashed arrows). This signal is likely due to the atoms of the underlying layer.

The above data allowed us to bring forward a model of the observed Si(001) surface reconstruction. This model takes into account the data of previous investigations carried out by different authors who suppose the $c(8 \times 8)$ structure to arise because of the presence of Cu atoms on the surface [5, 10, 13] as well as the results on low temperature deoxidation of the Si(001) surface according to which such process gives rise to $c(8 \times 8)$ structure formation as well [16].

The model is based on the following assumptions: (i) the outermost surface layer is formed by ad-dimers; (ii) the underlying layer has a structure of (2×1); (iii) every rectangular block consists of ad-dimers a number of which controls the block length.

Fig.5a shows a schematic drawing of the (8×8)-type structure (a unit cell is outlined). This structure is a basic one for the model brought forward. The elemen-

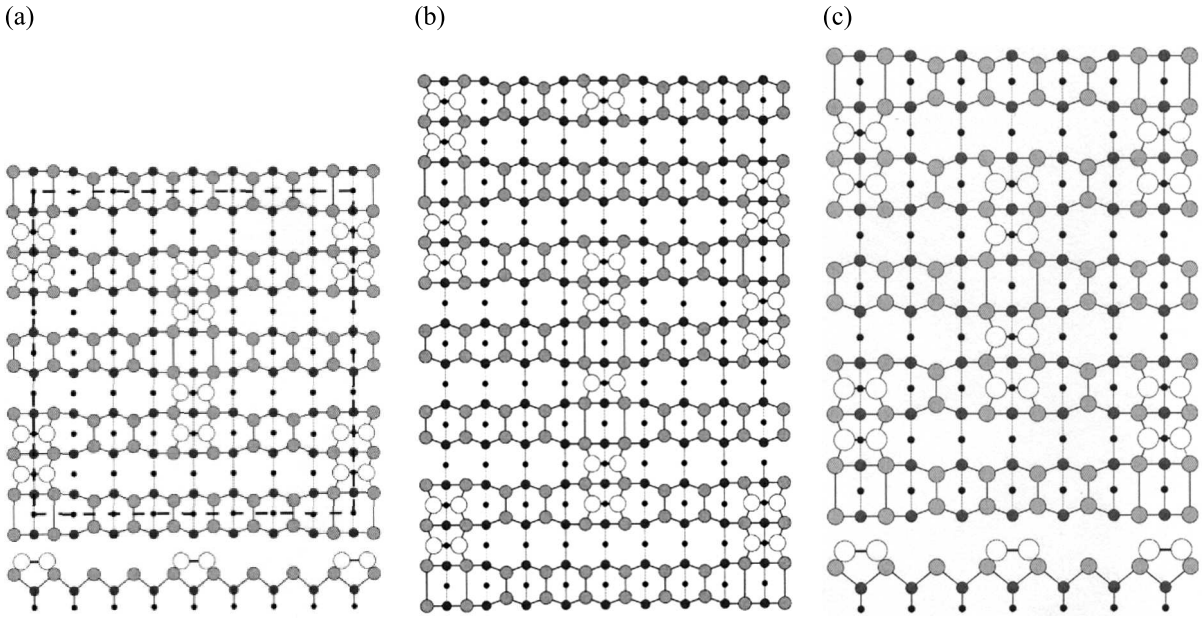

Fig.5. A schematic drawing of the $(8 \times n)$ structure. (a) (8×8) with the short blocks, a unit cell is outlined; (b) the same structure with the long block; (c) (8×6) structure

tary structural unit is a short rectangle. These blocks form raised rows running vertically (shown by empty circles). Smaller shaded circles show horizontal dimer rows of the lower terrace. The rest black circles show bulk atoms. Each rectangle consists of two couples of dimers separated with a dimer vacancy. The structures on the Si(001) surface composed of close ad-dimers are known to be stable [13]. In our model, a position of the rectangles is governed by the location of the dimer rows of the (2×1) structure of the underlying layer. The rows of blocks are always normal to the dimer rows in the underlying layer. Every rectangular block is bounded by the dimer rows of the underlying layer from both short sides.

Fig.5b demonstrates the same model for the case of the long rectangle. This block is formed due to the presence of an additional dimer in the middle of the rectangle. The structure consisting of one dimer is metastable [13], so this type of blocks cannot be dominating in the structure. Each long block is bounded on both short sides by the dimer rows of the underlying terrace, too. The presence of the long rectangle results in the formation of a vacancy defect in the adjacent row, this is shown in Fig.5b – the long block is drawn in the middle row, the dimer vacancy is present in the last left row.

According to our STM data the surface is disordered in the direction perpendicular to the rows of the blocks. The distances between the neighboring rows may be less than those in the (8×8) structure. Hence the structure presented in this paper may be classified as $(8 \times n)$ one. Fig.5c demonstrates an example of such a structure – a (8×6) one. Formation of the $(8 \times n)$ structure is explained by the diffusion of ad-dimers on the surface, and the diffusion along the dimer rows of the underlying layer being easier than across them.

An origin of this structure is not clear thus far. In the previous works studied the $c(8 \times 8)$ structure, authors attributed it to surface contamination by foreign atoms, e.g. by Cu [4–6, 10, 16]. This seems rather probable, but there is a circumstance that to some extent contradicts this viewpoint. The surface structure similar to that described in the current paper was formed on the samples subjected to a shorter annealing than that applied in this work and deoxidized only in part [1]. Moreover, the same structure was observed by us on the samples deoxidized in the UHV ambient by a flux of Si atoms at the specimen temperature around 770 °C [1]. The effect of the surface contamination by foreign atoms cannot be completely excluded, though.

In summary, it may be concluded that the Si(001) surface prepared under the conditions of the UHV MBE chamber in a standard wafer preparation cycle has $(8 \times n)$ reconstruction which is partly ordered only in one direction. Two types of unit blocks form the rows running along [110] and $[1\bar{1}0]$ axes. When the long block disturbs the order in a row a dimer-vacancy defect appears in the adjacent row in the vicinity of the long block to restore the chess-board order of blocks in the neighboring rows.

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