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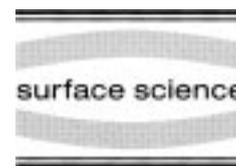
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Surface Science Letters

Pairs of Si atomic lines self-assembling on the b-SiC(100) surface: an 8×2 reconstruction

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Abstract

We investigate the transition between b-SiC(100) surface reconstructions using scanning tunneling microscopy (filled and empty electronic states). We find the unexpected formation of Si-dimer lines that are self-assembled by pairs with a long-range order resulting in an even 8×2 surface reconstruction. This very uncommon surface ordering results in the combination of alternating periodic 3×2 and 5×2 unit cells. In contrast to the b-SiC(100) 3×2 surface reconstruction, the Si-dimers are symmetric. Such atomic line pairs provide a novel type of one dimensional sub-nanostructure potentially having interesting characteristics. © 1998 Elsevier Science B.V. All rights reserved.

Keywords: Scanning tunneling microscopy; Silicon carbide; Superlattices; Surface relaxation and reconstruction; Surface structure, morphology, roughness and topography

Recent progress in silicon carbide (SiC) surface and interface experimental investigations has strengthened the already important fundamental as well as technological interests devoted to this promising refractory material. In fact, SiC is expected to bring rapidly an appropriate solution in high-power, high-frequency and high-temperature electronic devices and sensors [1,2]. Representing so far the only stable and long-range ordered form of the IV–IV compound family, SiC has two crystallographic phases, a hexagonal one

(a), the most studied but presenting numerous different polytypes, and a cubic one (b), more promising but difficult to grow [2]. To date, few detailed investigations have been performed on the b-SiC(100) surfaces due in part to important difficulties encountered in high-quality surface preparation [3–5]. Most of the knowledge is based on well-established techniques such as low electron energy diffraction (LEED), Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) [3–5]. It is only recently that advanced probes such as scanning tunneling microscopy (STM), medium energy ion scattering (MEIS) or synchrotron radiation related techniques have been used to probe b-SiC(100) surfaces [3–12]. Based on these studies, the b-SiC(100) surface is known to have several recon-

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structions [3–12]. These include: (1) Si-rich 3×2 and derived $(2n+1) \times 2$ series, (2) stoichiometric Si-terminated $c(4 \times 2)/2 \times 1$, (3) stoichiometric C-terminated $c(2 \times 2)$ and (4) C-rich 1×1 [3–12]. The $(2n+1) \times 2$ ($n \geq 1$) reconstructions, i.e. 3×2 , $5 \times 2, \dots$ were found to result from the long range ordering of Si dimer rows lying on a full Si plane [10]. These Si dimer rows, representing a total of $1/(2n+1)$ Si monolayer (ML), consist of Si dimers stacked perpendicularly to the row axis. Arranged with a lateral spacing of n times the surface lattice parameter ($a=3.08 \text{ \AA}$), these Si lines define the whole $(2n+1) \times 2$ ($n \geq 1$) reconstruction sequence, ultimately ending in the stoichiometric Si-terminated $c(4 \times 2)$ reconstruction ($n \rightarrow +\infty$). Since only odd Si-rich b-SiC(100) reconstructions have been observed so far, a 3×2 to $c(4 \times 2)$ transition model having no possible even reconstruction was proposed [10].

In this Letter, we provide evidence of a novel Si dimer row ordering pattern. This arrangement consists of long-range ordered Si-dimer lines that are self-assembled by pairs upon thermal annealing. This unexpected pair arrangement which, as far as we know, has never been observed before on any surface, occurs between the 3×2 and 5×2 surface orderings and results in an even 8×2 reconstruction of the b-SiC(100) surface. Contrary to the case of b-SiC(100) 3×2 , which only exhibits asymmetric dimers all tilted in the same direction [11], the Si dimers are symmetric.

The experiments were performed using an Omicron scanning tunneling microscope (STM) at working pressures of 4×10^{-11} Torr. The STM topographs (filled and empty electronic states) were recorded in the constant current mode, and were reproducible and not tip-dependent. Single-domain b-SiC thin films were prepared at LETI (CEA-Technologies Avancées) by chemical vapor deposition (C_3H_8 , SiH_4) growth on vicinal (4°) Si(100) surfaces. Surface preparation was achieved by annealing at 1150°C to remove native oxide as well as possible contaminants. Silicon surface stoichiometry was restored by room-temperature Si deposition followed by brief annealing at 1000°C . Increasing annealing times allowed the complete sequence of b-SiC(100) reconstructions to be obtained, ranging from Si-rich 3×2 to C-rich 1×1

[3–12]. The surface quality was checked by LEED to exhibit sharp diffraction patterns. Other experimental details can be found elsewhere [11,12].

We first show in Fig. 1 the effect of thermal annealing sequences on the b-SiC(100) 3×2 surface structure using STM (filled electronic states). Fig. 1a shows a $150 \text{ \AA} \times 150 \text{ \AA}$ topograph of the Si-rich 3×2 reconstruction exhibiting dimer rows with the characteristic oval shape spots associated with silicon dimers [11]. Fig. 1b and c show representative STM topographs of the 3×2 surface modifications (Fig. 1a) after short and longer annealing times at 1150°C . On the latter STM images, one can clearly observe that annealing results in Si atom removal leading to the formation of straight parallel lines arranged in a more or less dense pattern. In Fig. 1b, two different types of line ordering are observed. The first one includes a dense array (marked A in the margin) with a lateral distance between lines of about 9.2 \AA , i.e. $3a$ (a being the surface lattice parameter) and corresponds to a remaining 3×2 reconstruction area. The second one is made of a less dense array where lines are very interestingly set by pairs that are regularly spaced on the surface. The effect of further annealings can be traced in Fig. 1c, which exhibits two different types of line patterns with, as in Fig. 1a and b, some dimer vacancies. Indeed, we observe the previously depicted line pair arrangement (marked B) together with another and less dense single line arrangement. In the latter case, the measured lateral distance between lines close to 15.4 \AA ($5a$) indicates clearly a 5×2 reconstruction area [10]. The two latter STM topographs suggest the existence of a new b-SiC(100) surface ordering coexisting with the 3×2 (Fig. 1b) and 5×2 (Fig. 1c) reconstructions. As a matter of fact, upon annealing, part of the top silicon atom desorption leads to the formation of less dense Si dimer row reconstructions having odd $(2n+1) \times 2$, $n \geq 1$, LEED patterns that ultimately results in the formation of single self-organized Si atomic lines as recently observed [13].

In order to explore the long-range order of this interesting and novel surface structure, we performed LEED measurements. As can be seen in Fig. 2a, an even 8×2 LEED pattern is precisely observed after annealing the 3×2 surface and

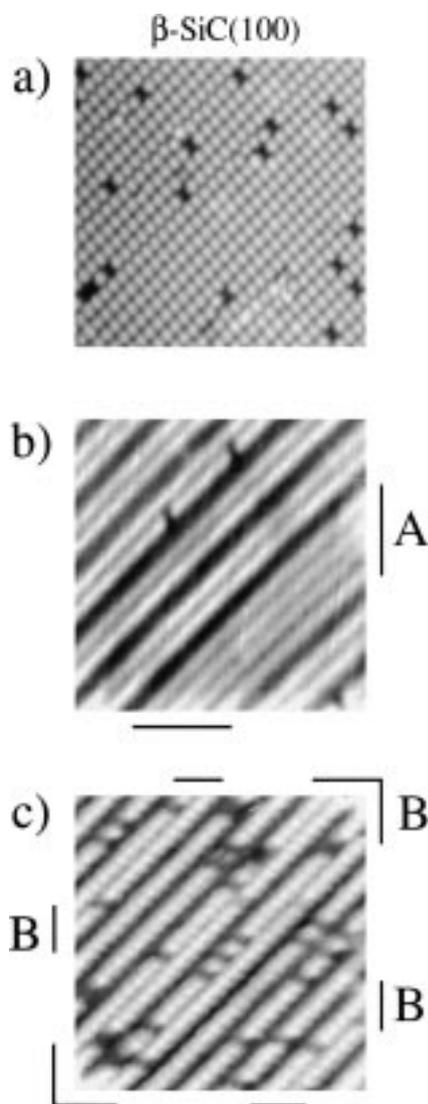


Fig. 1. (a) b-SiC(100) 3×2 surface: $150 \text{ \AA} \times 150 \text{ \AA}$ STM topograph (filled electronic states). The tip bias is $V_t = +2.0 \text{ V}$, and the current $I_t = 0.2 \text{ nA}$. (b) $150 \text{ \AA} \times 150 \text{ \AA}$ STM topograph (filled electronic states) of the surface after a short thermal annealing of the b-SiC(100) 3×2 surface at $T = 1150^\circ\text{C}$. Region A marked by black lines corresponds to a 3×2 -like pattern. Non-marked areas show atomic line pair ordering. The tip bias is $V_t = +2.82 \text{ V}$, and the current $I_t = 0.33 \text{ nA}$. (c) $150 \text{ \AA} \times 150 \text{ \AA}$ STM topograph (filled electronic states) of the surface after a long thermal annealing of b-SiC(100) 3×2 surface at $T = 1150^\circ\text{C}$. Regions marked B indicate the presence of atomic line pairs. Non-marked areas exhibit a 5×2 -like pattern. One can also notice the presence of dimer vacancies. The tip bias is $V_t = +2.61 \text{ V}$, and the current $I_t = 0.49 \text{ nA}$.

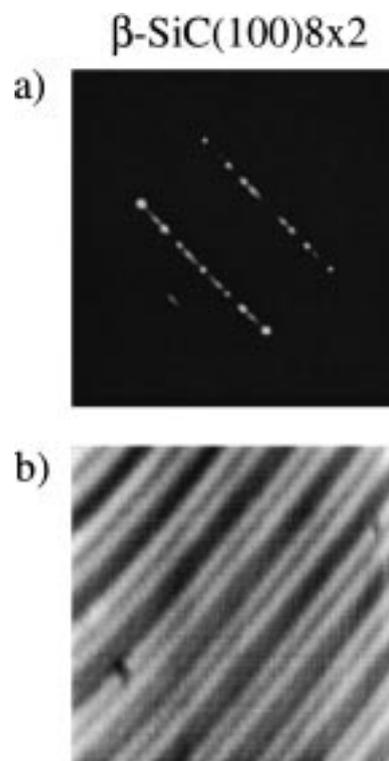


Fig. 2. (a) 8×2 LEED photograph ($E_p = 89 \text{ eV}$) of the b-SiC(100) atomic line pair reconstruction. (b) $150 \text{ \AA} \times 150 \text{ \AA}$ filled states STM topograph of the atomic line pair b-SiC(100) reconstruction. Tip bias is $V_t = +3.55 \text{ V}$ at a current $I_t = 0.33 \text{ nA}$.

before the 5×2 reconstruction appearance. This demonstrates the long-range ordering by pairs of these Si dimer lines in a novel 8×2 surface reconstruction. The $150 \text{ \AA} \times 150 \text{ \AA}$ STM topograph (filled electronic states) displayed in Fig. 2b shows a homogeneous array of dimer pairs only, which further stresses that this very particular surface ordering is not just a transient phase, but a full 8×2 surface reconstruction existing by itself.

To obtain a deeper structural insight for these dimer row pairs, we now look at STM topographs by probing and comparing both filled and empty electronic state topographs. Fig. 3a and b show $100 \text{ \AA} \times 100 \text{ \AA}$ topographs of the b-SiC(100) 8×2 surface atomic line pairs, which were obtained by tunneling into the filled and empty states, respectively. In Fig. 3a (filled states), Si dimer row pairs are regularly set in a periodic arrangement with

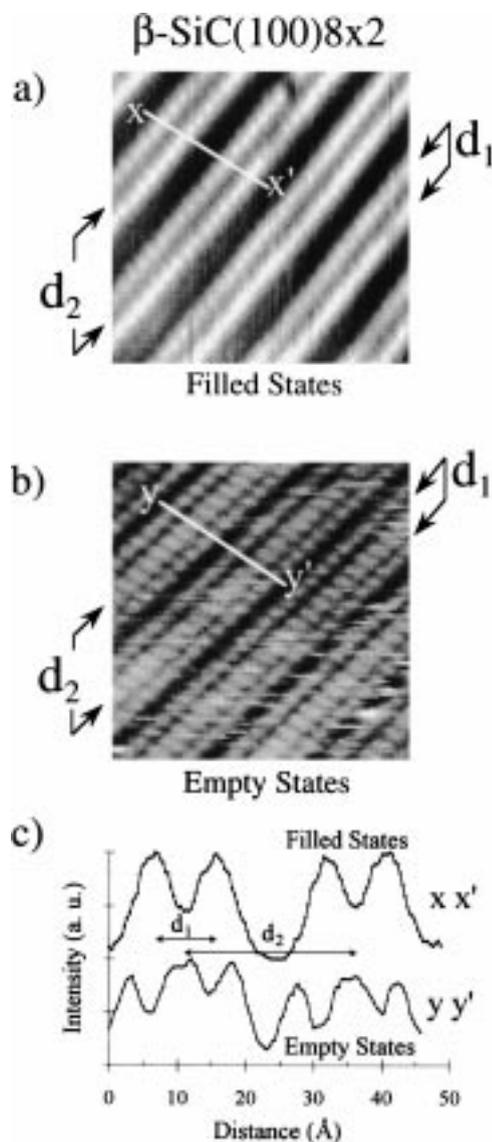


Fig. 3. (a) $100 \text{ \AA} \times 100 \text{ \AA}$ filled electronic state STM topograph of Si atomic line pairs on b-SiC(100). The intra-pair distance d_1 represents the lateral row-to-row distance within an atomic line pair. The inter-pair distance d_2 represents the distance between the centers of two neighboring atomic line pairs. The tip bias is $V_t = +3.55 \text{ V}$ at a current $I_t = 0.33 \text{ nA}$. (b) $100 \text{ \AA} \times 100 \text{ \AA}$ empty states STM topograph of Si atomic line pairs on b-SiC(100) reconstruction. Tip bias is $V_t = -2.75 \text{ V}$, and current $I_t = 0.33 \text{ nA}$. d_1 and d_2 are as in (a). (c) Height profiles covering two line pairs along (1) xx' in (a) (filled states) and (2) yy' in (b) (empty states).

the characteristic oval shape spot associated with independent Si dimers. Within a line pair, the line-to-line distance labeled as intra-pair distance d_1 (Fig. 3a) is $9 \pm 1 \text{ \AA}$. The inter-pair distance d_2 (Fig. 3a) measuring the distance between the centers of two adjacent pairs is $25 \pm 1 \text{ \AA}$. The dimer-to-dimer distance within a line is $5.8 \pm 0.9 \text{ \AA}$, as for the 3×2 surface reconstruction [11]. The intra-pair distance d_1 is close to three times the surface lattice parameter a (3.08 \AA), whereas the inter-pair distance d_2 is close to $8a$, and the dimer to dimer distance d_2 approaches $2a$. The latter distance is equivalent to that observed for the odd $(2n+1) \times 2$ reconstructions and therefore supports the Si dimer stacking rows as being the common features of all Si-rich b-SiC(100) $(2n+1) \times 2$ reconstruction types. This means that Si dimer lines belonging to the same pair have the same position as for the 3×2 reconstruction. Considering two neighboring line pairs, the closest lateral line-to-line distance scales at $8-3=5$, i.e. five times the surface unit cell parameter a as for the 5×2 reconstruction. Thus, the periodicity of this line pair reconstruction is $3a+5a=8a$ and $2a$ perpendicular and parallel to the dimer rows respectively, in excellent agreement with the even 8×2 LEED pattern (Fig. 2a).

We now turn to the empty electronic states STM topograph (Fig. 3b), which exhibits a slightly more complicated picture. One can observe large straight bands subdivided into three parts: a large central line surrounded by two narrower ones. Two adjacent central lines are separated by the same distance as that between two adjacent row pairs in the filled state image, i.e. one inter-pair distance $d_2 = 25.3 \pm 0.6 \text{ \AA}$. The intra-pair distance d_1 corresponds to the distance between the two adjacent subdividing dark gaps within a band.

To understand the origin of such a topograph, we determined out height profiles perpendicular to the line pairs for both filled (Fig. 3c, top) and empty (Fig. 3c, bottom) electronic states. Contrary to the b-SiC(100) 3×2 case, which clearly exhibits two features [11], no such splitting is observed here for an empty-state topograph along a dimer pair. As a matter of fact, for the 3×2 reconstruction, each Si dimer oval splits into two smaller spots, which results from tunneling into the empty dangling bond orbitals associated with individual

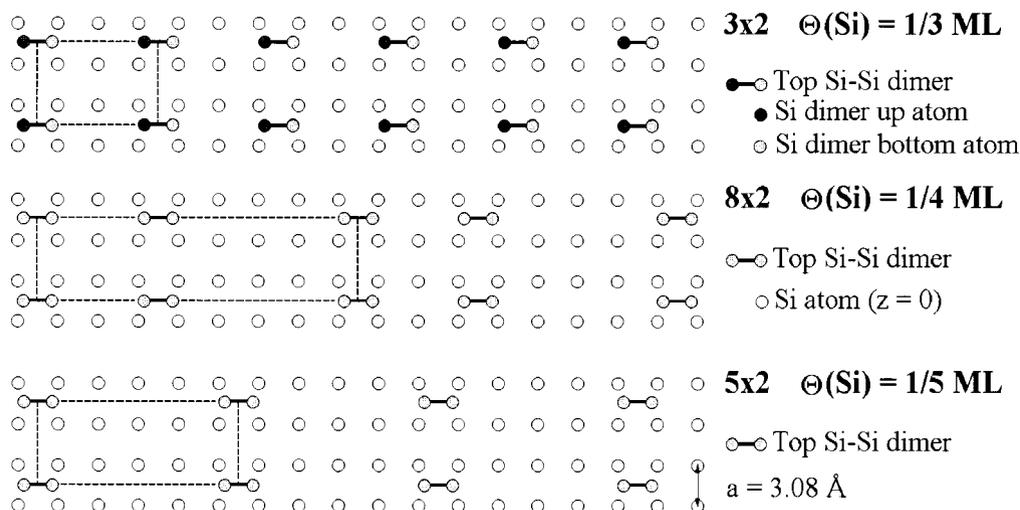


Fig. 4. Schematic structure of the b-SiC(100) 8×2 Si dimer line ordering pattern together with those of the 3×2 and 5×2 reconstructions. Dashed lines represent corresponding unit cells. $H(\text{Si})$, the Si coverage is also indicated. Note that the 3×2 reconstruction has parallel asymmetric Si-dimers (PAS) all tilted in the same direction [11], unlike the 8×2 and 5×2 reconstructions, which exhibit symmetric dimers. The underlying Si plane is represented unreconstructed.

Si atoms forming a dimer [11]. For the 3×2 reconstruction, the Si dimers were found to be asymmetric and all tilted in the same direction, which seems to facilitate identification of the two Si atoms' contributions [11]. The height profile displayed in Fig. 3c perpendicular to the Si dimer rows exhibits a regular shape, which indicates that the two Si atoms are at the same height, in contrast to the b-SiC(100) 3×2 reconstruction where dimers are asymmetric [11]. This dimer symmetric character for the line pairs is likely to result, in the middle part of the dimer row pair, in a significant overlap between dangling bond orbitals that would in turn give an unresolved central line, as observed in Fig. 3c. In addition, this empty-state topograph stresses that the dimer direction is perpendicular to the row axis as shown for the 3×2 reconstruction [11]. This picture, which, once again [11], is based on direct identification of Si atom position, rules out previous suggestions [10] of dimer direction parallel to the dimer row.

Our present STM results indicate that, in addition to the known single dimer row reconstruction mode, the b-SiC(100) surface undergoes a novel reconstruction based on an interesting Si dimer line pair array. This unique Si-dimer pair arrangement takes place between the b-SiC(100) 3×2 and

5×2 surface reconstructions and results in an unexpected even 8×2 reconstruction. Fig. 4 displays a schematic view of this novel b-SiC(100) 8×2 surface reconstruction together with the 3×2 and 5×2 reconstructions for the sake of comparison. The 8×2 reconstruction consists of dimer line pairs made from stacked dimers having a direction perpendicular to the line axis. Within one line pair, the dimer rows are separated by $3a$, as for the 3×2 reconstruction. The closest distance between two lines belonging to two different pairs ($5a$) corresponds to a 5×2 periodicity. From a structural point of view, the line pair reconstruction can be seen as a mixture of 3×2 and 5×2 unit cells. Such a structural array gives a Si coverage, H , of $1/4 \text{ ML}$, intermediate between $1/3 \text{ ML}$ for the 3×2 [11] and $1/5 \text{ ML}$ associated with the 5×2 . Therefore, this new line pair reconstruction, which is derived from the 3×2 surface reconstruction by selective Si atom organization upon thermal annealing, is not only able to coexist with the Si-rich 3×2 and 5×2 surface reconstructions, but also appears as a novel intermediate structure between the two latter. It demonstrates that the only constraint about dimer line lateral position is that the closest distance between two neighboring dimer rows has to be an odd multiple of the a

surface parameter, which, contrary to previous understanding [10], allows even $(2n) \times 2$ reconstructions since the sum of two odd numbers always gives an even number.

Finally, one can anticipate that, due to their unique structure, these Si atomic line pairs would become an ideal system for metal or molecule (inorganic and organic) adsorption studies. In this context, such a low-dimensional structure surface may show selective and/or anisotropic metallization or passivation upon appropriate adsorbate deposition. This could lead to the discovery of nanostructures having novel and interesting characteristics.

In conclusion, using scanning tunneling microscopy, we provide evidence of a novel 8×2 surface reconstruction based on the formation of unexpected Si atomic line pairs. This even b-SiC(100) 8×2 surface reconstruction takes place at the transition between the 3×2 and 5×2 reconstructions. Unlike the 3×2 surface reconstruction, these Si dimer lines are composed of symmetric Si-dimers. Such atomic lines that self-organize by pairs have never been observed for other surfaces.

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