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**Atomically-resolved Structure and Bonding of Delta-doped
Boron Layers on Si(001)**

by

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Atomically Resolved Structure and Bonding of Delta-doped Boron Layers on Si(001)

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Abstract

Scanning tunneling microscopy has been used to study the formation of "delta-doped" layers by thermal decomposition of diborane on Si(001). STM images reveal a number of boron-induced reconstructions which arise from ordered arrangements of three structural subunits. Based on the symmetry of the STM images and the bonding locations of the observed features with respect to the Si(001) lattice, a structural model is proposed with accounts for the observed STM features. The principal structural subunit is shown to be an ordered arrangement of four boron atoms at substitutional sites in the first bulk-like silicon layer, which is then capped with ordered arrangements of silicon dimers and dimer vacancies.

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Doping of semiconductor structures is well known to be a strongly non-equilibrium process involving a complex interplay of surface structure and chemical kinetics. Recent investigations¹⁻⁶ have shown that the unique interfacial chemistry of boron with silicon permits the fabrication of narrow, non-equilibrium two-dimensional layers of highly-doped silicon with high structural and electrical quality. Even though the local boron concentration in these "delta-doped" layers exceeds the bulk solubility limit by more than two orders of magnitude, deposition of silicon atop the layers results in crystalline, epitaxial growth, thereby permitting the fabrication of buried, atomically-sharp layers of electrically-active dopants.^{3, 7, 8}

The growth of such non-equilibrium structures by chemical techniques presents a novel method for preparation of nanometer-scale electronic devices based on self-limiting interfacial chemistry. In this study, we report the first scanning tunneling microscopy (STM) studies investigating the atomic-scale structure of delta-doped boron layers produced by thermal decomposition of diborane. Our results show that boron interacts with Si(001) to produce several complex, ordered reconstructions which can be described as arrangements of three basic structural subunits. Our results provide new insight into the structure and bonding within these delta-doped layers.

Samples consisted of 5mm x 2 cm pieces of commercial Si(001) wafers (Wacker Chemitronic). These pieces were cleaned by annealing at 1425 Kelvin in ultrahigh vacuum⁹ and, after cooling to room temperature, were imaged with the STM. These well-characterized surfaces were then exposed to 5000 Langmuirs of 1% diborane in He at 815 Kelvin followed by annealing at 1000 Kelvin for 90 seconds, producing the surface structures described here. Other experiments show that these same features are observed over a wide range of temperatures and exposures. At very high exposure and high temperatures formation of amorphous surface layers was observed.

While the clean Si(001) surface prepared by high-temperature annealing is characterized by large, flat terraces separated by monatomic steps, fig. 1a shows that exposure to diborane at elevated temperatures produces a large number of islands atop the terraces. Additionally, many regions of the original

substrate terrace appear ~ 0.3 Å lower than the rest of the (2x1) terrace, giving the overall surface a speckled appearance. At higher resolution, fig. 1b shows that the majority of the islands exhibit a complex boron-induced reconstruction, although some small regions of the islands show the features characteristic of the clean, (2x1)-reconstructed Si(001) surface. Many small areas of the substrate terrace also show new boron-induced reconstructions; approximately 1/3 of these boron-induced reconstructed substrate ("RS") regions can be observed.

The formation of islands atop the extended Si(001) terraces requires ejection of silicon from the substrate plane. The STM images show that boron-induced reconstructions occupy almost all of the area of the islands and many smaller regions of the original terrace having nearly the same total area. Although the reconstruction may involve more than one atomic layer, this measurement indicates that the ratio of the average volume density of silicon atoms in the reconstructed regions $\rho_{\text{reconstructed}}$ to the bulk Si atom density ρ_{bulk} is given approximately by $\frac{\rho_{\text{reconstructed}}}{\rho_{\text{bulk}}} \approx \frac{2N-1}{2N}$, where N is the number of layers involved in the reconstruction.

Figure 2 shows high-resolution images of the boron-induced reconstructions. In fig. 2a, small regions showing c(4x4) symmetry (diamond-shaped unit cell, labeled "α") and (4x4) symmetry (square unit cell, labeled "γ") can be observed. Additionally, some regions showing the (2x1) reconstruction of the "clean" Si(001) surface can be observed in the lower left and upper left corners (rectangular unit cell, labeled "Si"). Further insight into these reconstructions is obtained by comparing high-resolution STM images acquired over a single region at both negative sample bias (probing occupied electronic states) and positive sample bias (probing unoccupied states). In fig. 2b (-2V bias) and 2c (+2V bias), the surface again shows primarily the c(4x4) reconstruction, although a few unit cells of (4x4) symmetry are again observed. Additionally, at the lower right of fig. 2b and 2c, small regions exhibiting yet another reconstruction of c(4x4) symmetry can be observed, labeled "β". For clarity, we will refer to the most common structure of c(4x4) symmetry as "α-c(4x4)", the less common c(4x4) reconstruction as "β-c(4x4)", and the (4x4) reconstruction as "γ-(4x4)".

Close inspection of a large number of images indicates that all three boron-induced reconstructions share common structural features. We consider first the most common, the α-c(4x4) reconstruction. At negative sample bias each unit cell contains two equivalent oval-shaped protrusions and a single, unique oval protrusion which under these imaging conditions appears 0.35 Å higher. Although the highest-symmetry unit cell can be described as a single high oval protrusion bounded on each side by a single lower oval, examination of the reconstruction at the top and bottom edges of the island in fig. 2b and 2c shows that the edges of reconstructed areas always terminate with a pair of the lower protrusions. Thus, the α-c(4x4) reconstruction is best described in terms of two structural building blocks: the first is pair of two equivalent oval-shaped protrusions, labeled "A" in fig. 2. The second is the single unique oval-shaped protrusion, labeled "B" in fig. 2. This structure consisting of one "A" subunit and one "B" subunit repeats every 4a₀ along the $\langle 1\bar{1}0 \rangle$ direction and is ordered between adjacent rows to form a structure with local c(4x4) symmetry as indicated by the unit cell drawn in fig. 2. At positive bias (tunneling into unoccupied surface states), fig. 2c shows that the "B" feature appears to be split into two well-resolved nearly circular protrusions, and the two oval-shaped protrusions constituting the "A" feature now appear 0.4 Å higher than the "B" feature, but otherwise unchanged in appearance.

The γ-(4x4) reconstruction is nearly identical to that described above, except that the "B" feature is absent in half the unit cells. Images at both positive and negative bias reveal what appears to be a vacancy at its expected position.

Finally, the β-c(4x4) reconstruction visible at lower right in fig. 2b and 2c appears to arise from an ordered arrangement of the "A" features alone. Within each β-c(4x4) unit cell, the two ovals are separated along the $\langle 1\bar{1}0 \rangle$ direction by a distance which alternates between 5.0 Å and 10.4 Å (compared with the Si(001) surface lattice constant of 3.84 Å), such that the ovals show a clear grouping into pairs. The shape, apparent grouping of these oval protrusions, and measurements of their apparent height as a function of applied bias all correlate almost exactly with our observations for the "lower" oval protrusions observed in the α-c(4x4) and β-c(4x4) reconstructions.

indicating that the "A" subunit is common to all three boron-induced reconstructions.

To develop a model for the surface structure, it is necessary to consider the primary structural subunit of the Si(001) surface, which is the Si=Si dimer. The small regions of "clean" Si(001)-(2x1) visible in fig. 2b and 2c show that at negative sample bias each Si=Si dimer of the clean Si(001) surface gives rise to a single oval-shaped protrusion, while at positive bias images each dimer gives rise to two well-resolved protrusions. This change is well understood^{10, 11} to arise from the differing spatial distributions of the occupied π and unoccupied π^* orbitals which are the dimer electron states nearest the Fermi energy.¹² STM images of the α -c(4x4) and γ -(4x4) reconstructions in fig. 2b and 2c shows that the central oval-shaped protrusion within each unit cell changes with bias in a manner nearly identical to that of the clean Si(001) dimers, suggesting a similar structural origin.

Based on detailed analysis of the STM images in conjunction with other experimental evidence, we have developed models for the structural subunits and their ordering to form the boron-induced reconstructions as shown in figure 3. Previous studies of this system have established several important facts. First, several investigators have shown that formation of the "δ-doped" structures is optimized at a coverage of 0.5 monolayer^{3, 8, 13} (where 1 monolayer = 6.8×10^{14} cm⁻² boron atoms). Low-energy electron diffraction studies^{1, 3-5} have suggested that the δ-doped layers are ordered with a (2x1) periodicity, although Sardola, et al.¹⁴ reported a (2x2) periodicity at low boron coverage. Hall effect measurements³ have shown that nearly 100% of this boron is electrically active, demonstrating that the boron atoms occupy substitutional sites in the Si lattice. Finally, transmission electron microscopy, ion scattering, ion channeling, and x-ray studies^{2, 3, 7, 8} have shown that high-quality epitaxial silicon films can be grown atop these δ-doped layers at low temperatures, and that the buried boron layers retain their substitutional location and extremely narrow depth distribution. This remarkable ability indicates that the outermost layer of silicon atoms is not drastically different from that of a bulk-terminated or reconstructed Si(001) surface, so that deposition of Si on the δ-doped layer results in continued high-quality crystal growth.

The "A" feature indicated in fig. 2 is common to all three boron-induced reconstructions. The two oval-shaped features in each of these subunits appear to be dimers; however, they have a different apparent height than those of the clean surface, they fail to show the splitting at positive sample bias that is characteristic of the clean Si(001) surface, and they are always observed in pairs. Based on these observations and the bonding location symmetry with respect to the underlying Si(001) lattice, we propose that they arise from Si-Si dimers bonded above a Si(001) surface in which 50% of the Si atoms in the first bulk-like layer are replaced by boron at the positions indicated in black in fig. 3. Since boron is smaller than silicon, surface Si dimers would be geometrically distorted toward the boron atoms; this geometric effect and the accompanying asymmetry in electronic properties would make the apparent spacing between dimers a non-integral multiple of a_0 (≈ 3.84 Å), in agreement with our experimental measurements.

The "B" feature exhibits strong changes in appearance with applied bias demonstrating a strong spatial separation of occupied and unoccupied electronic states. However, over the entire range of bias voltages investigated these changes are *identical* to those observed for Si=Si dimers of the clean surface. The bonding location of feature B is also consistent with its interpretation as a Si=Si dimer. Thus, we propose that the "B" feature arises from a Si=Si dimer bonded to four second-layer Si atoms in a local environment similar to that of the clean Si(001) surface.

All three observed reconstructions can be described as ordered combinations of the "A" and "B" structural subunits together with dimer vacancies. The common α -c(4x4) reconstruction arises from one "A" subunit and one "B" subunit in each unit cell, as depicted in fig. 3. The β -c(4x4) reconstruction arises from one "A" subunit and one dimer vacancy in each unit cell, while the γ -(4x4) reconstruction is obtained by replacing only half of the "B" subunits (Si=Si dimers) with dimer vacancies, leading to a larger unit cell containing two "A" subunits, one "B" subunit, and one dimer vacancy arranged as shown in fig. 3.

The most common reconstruction, the α -c(4x4) structure, is likely the principal boron-induced structure for δ-doping of boron in

silicon. In this structure, the boron coverage predicted by our model is $3.4 \times 10^{14} \text{ cm}^{-2}$, or 0.5 monolayer, in good agreement with previous measurements.^{3-6,15} While previous studies have shown a (2x1) LEED pattern,^{1, 3-5} our studies show that the surface unit cells are actually larger, but domain boundaries and the other coexisting B-induced reconstructions destroy the long-range coherence, leaving only the short-range (2x1) Fourier components.^{1, 3-5} The three dimers in the topmost layer and the four Si atoms in the next (boron-containing) layer give an overall Si atom density in the two-layer reconstruction which is 5/8 of the bulk Si atom density. This is in good agreement with our observation that the islands and reconstructed terraces have nearly equal areas, which for a reconstruction extending two layers deep implies an average Si atom density in the reconstructed regions which is 3/4 that of the bulk.

In all three reconstructions, it is likely that Si atoms exposed in the second layer will minimize their energy through π -bonding interactions, in a manner similar to that predicted by Pandey in his π -bonded vacancy defect model for Si(001).¹⁶ Since the existence of π -bonding interactions is strongly supported both theoretically^{16, 17} and experimentally⁹ we have included these bonding interactions in the models in figure 3.

The silicon dimers forming the "A" subunits appear significantly different from those of the clean Si(001) surface. We attribute this difference to a combination of structural and electronic effects. Electronic structure calculations¹⁸ for SiH_3BH_2 , $(\text{SiH}_3)_2\text{BH}$, and $(\text{SiH}_3)_3\text{B}$ predict Si-B bond lengths of $2.04 \pm 0.01 \text{ \AA}$, substantially shorter than the $2.41 \text{ \AA}$ bond length between a dimerized silicon atom and the underlying Si substrate at the clean Si(001) surface¹⁹ or the $2.35 \text{ \AA}$ bond length in bulk silicon. X-ray structural analysis of silicon-capped δ -doped B/Si(001) layers shows a contraction of 0.45 $\text{\AA}$ along the $\langle 001 \rangle$ direction,⁷ and both x-ray diffraction measurements and electronic structure calculations of the related $\text{Si}(111)-(\sqrt{3} \times \sqrt{3})$ reconstruction show that the B-Si bond length is between 2.0 and 2.1 $\text{\AA}$.^{20, 21} These observations suggest that B substitution in the Si(001) surface will significantly distort the Si lattice to shorten the B-Si bond.

The observation that δ -doped boron layers are electrically active indicates that the boron atoms in the "A" structure are negatively charged. Our STM images of these dimers fail to show the characteristic "splitting" at positive bias, suggesting that the π -bond is indeed broken but the σ bond remains intact. Breaking the dimer π -bond allows the dimer Si atoms to move closer to the boron atoms and also allows the electrons formerly in the π bond to transfer to the B atoms. This charge transfer leaves the boron atoms negatively charged with three B-Si bonds and a single electron in a fourth p-like orbital, and simultaneously eliminates the "dangling bond" π state of the Si-Si dimers. Silicon-to-boron electron transfer is further indicated in STM images from a comparison of the apparent height of the π -bonded Si=Si dimers of the "B" subunit with the silicon dimers of the "A" subunit; the latter show a lower density of occupied states and an increased density of unoccupied states. A similar charge transfer has been reported in studies of the boron-induced $\text{Si}(111)-(\sqrt{3} \times \sqrt{3})$ reconstruction,^{21, 22, 23} in which adatom "dangling bond" states were shown to transfer charge to the boron atom, decreasing the density of filled states and increasing the density of empty states at the adatom sites.

Finally, we note that since this bonding picture predicts that adjacent B atoms will each have a single electron in a p-like orbital, it is also likely that the adjacent B atoms have at least some bonding interaction, making the B atoms effectively four-fold coordinated. Electronic structure calculations should provide further insight into the nature of boron-boron and boron-silicon bonding in these δ -doped layers.

In summary, we have shown that the interaction of boron with silicon to produce " δ -doped" layers produces a number of coexisting ordered reconstructions. We have used STM images to reveal the electronic states, local symmetry and crystallographic locations of the observed features with respect to the underlying Si(001) lattice. Using this information we have developed a structural model which accounts for the observed STM images of the delta-doped surface layers. The principal structural unit is an ordered arrangement of four boron atoms at substitutional sites in the first bulk-like silicon layer. This layer is then capped with ordered arrangements of silicon dimers and dimer vacancies.

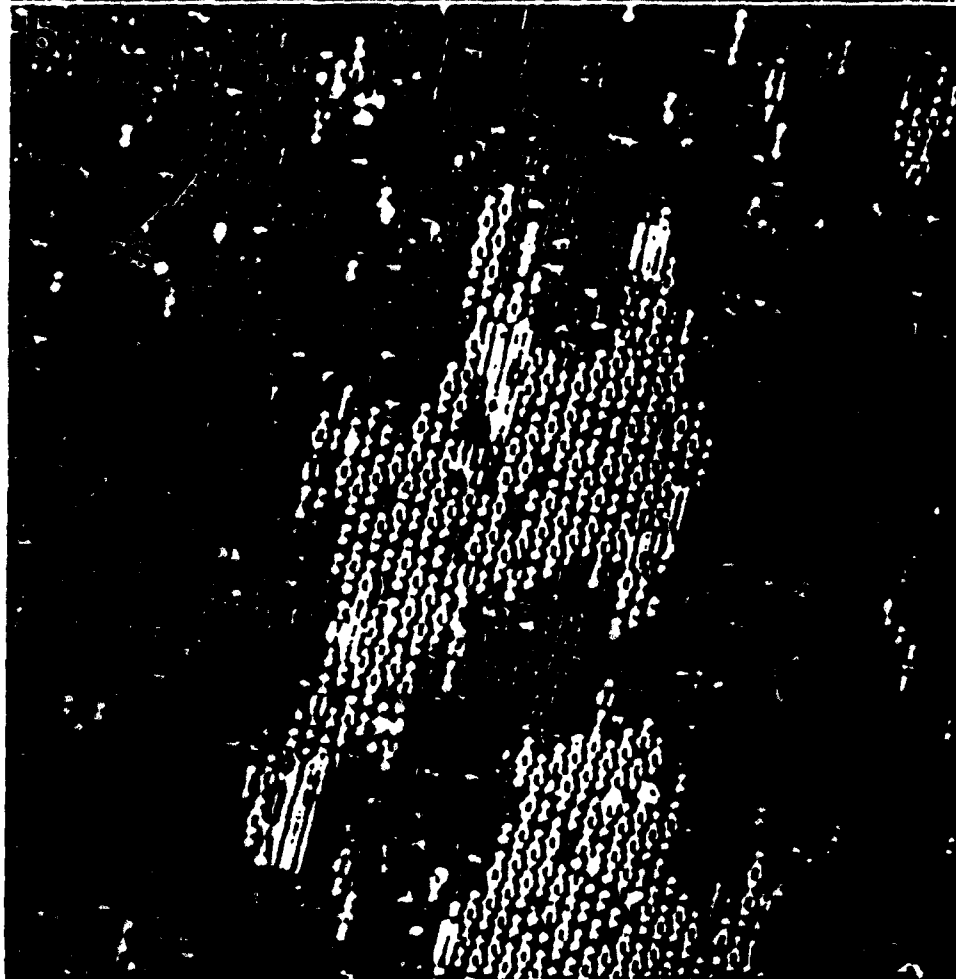
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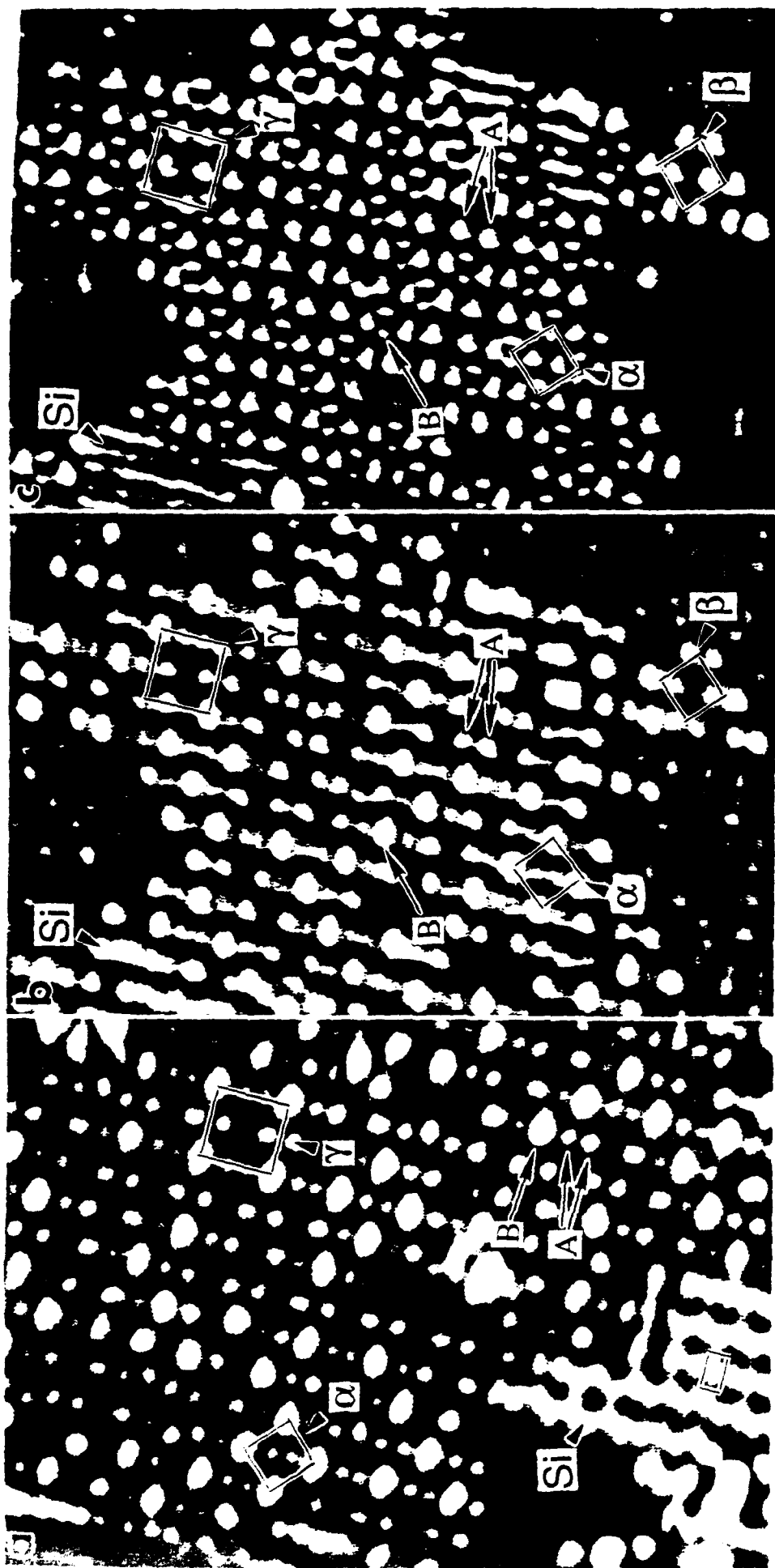
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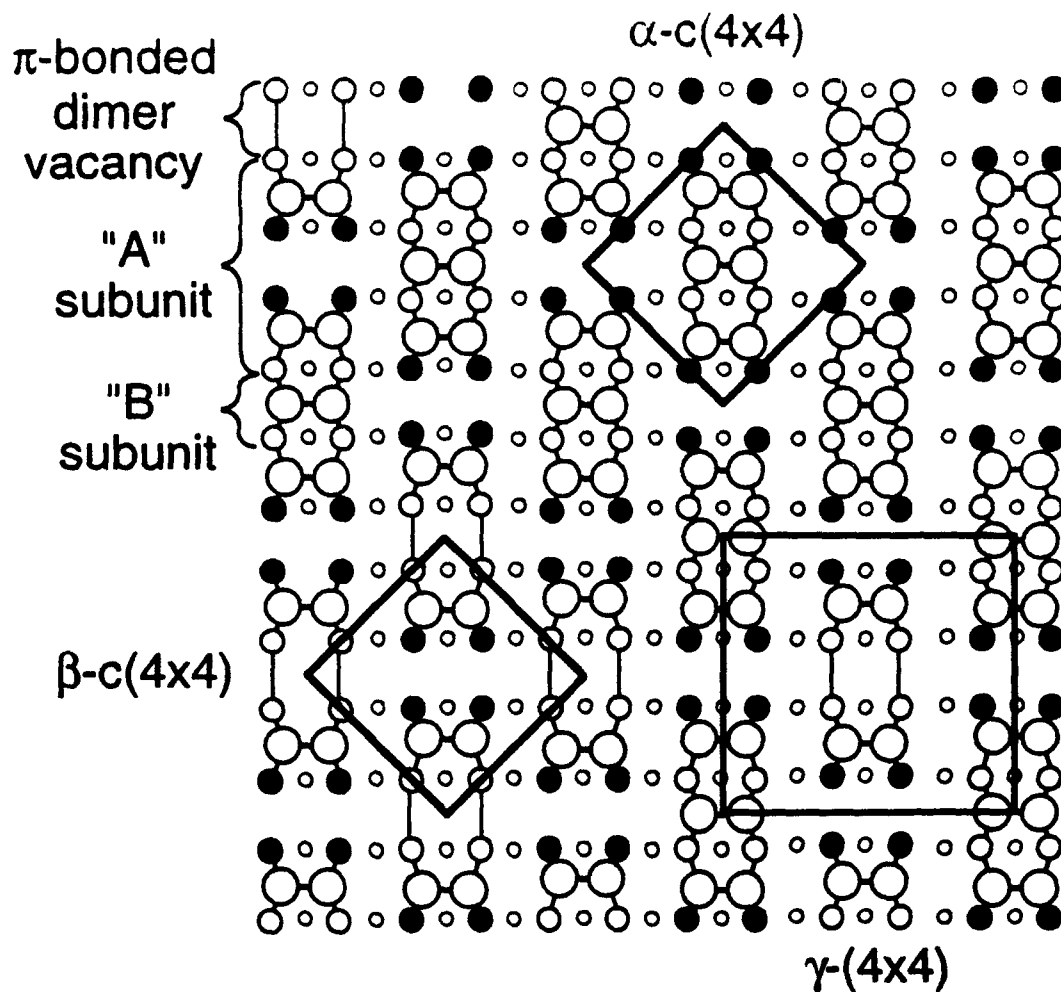
Figure Captions:

- Fig. 1a: Large-scale STM image of Si(001) surface after exposure to diborane as described in text. Image dimensions $1700 \text{ \AA} \times 1700 \text{ \AA}$, $V_{\text{sample}} = -1.6 \text{ V}$; $I_{\text{tunnel}} = 0.3 \text{ nA}$.
- Fig. 1b) STM image of diborane-exposed surface showing formation of reconstructed islands and patchy reconstruction of substrate terrace. $V_{\text{sample}} = +2.0 \text{ V}$; $I_{\text{tunnel}} = 0.2 \text{ nA}$.
- Fig. 2: High-resolution images of boron-induced reconstructions on Si(001) surface. α , β , and γ denote α -c(4x4), β -c(4x4), and γ -(4x4) unit cells of observed reconstructions; A and B denote structural subunits of reconstructions, as described in text..
- a) High-resolution image showing boron-induced reconstruction and region of clean Si(001)-(2x1), with (2x1) unit cell outlined. Image area $108 \text{ \AA} \times 152 \text{ \AA}$; $V_{\text{sample}} = -2.0 \text{ V}$; $I_{\text{tunnel}} = 0.2 \text{ nA}$.
- b) High-resolution imaging of occupied surface electronic states. Image area $104 \text{ \AA} \times 157 \text{ \AA}$; $V_{\text{sample}} = -2.0 \text{ V}$; $I_{\text{tunnel}} = 0.2 \text{ nA}$.
- c) Image showing identical region as (b), but tunneling into unoccupied electronic states. Image area $104 \text{ \AA} \times 157 \text{ \AA}$; $V_{\text{sample}} = +2.0 \text{ V}$; $I_{\text{tunnel}} = 0.2 \text{ nA}$.
- Fig. 3: Structural sub-units of boron-induced reconstructions, and proposed arrangements of these subunits to form α -c(4x4), β -c(4x4), and γ -(4x4) unit cells.



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Fig.





-  Top-layer silicon atoms (as dimers)
-  Second-layer silicon atoms
-  Second-layer boron atoms
-  Third-layer silicon atoms