Atomically accurate Si grating with 5.73 nm period

A. Kirakosian
Department of Physics, University of Wisconsin Madison, Madison, Wisconsin 53706

R. Bennewitz
Department of Physics and Astronomy, University of Basel, 4056 Basel, Switzerland

J. N. Crain
Department of Physics, University of Wisconsin Madison, Madison, Wisconsin 53706

Th. Fauster
Lehrstuhl für Festkörperphysik, Universität Erlangen-Nürnberg, D-91058 Erlangen, Germany

J.-L. Lin, D. Y. Petrovykh, and F. J. Himpsel[a)]
Department of Physics, University of Wisconsin Madison, Madison, Wisconsin 53706

(Received 8 May 2001; accepted for publication 12 July 2001)

A vicinal surface of silicon is found that exhibits an atomically accurate step pattern with a period of 5.73 nm, corresponding to 17 atomic rows per (111) terrace. It can be viewed as reconstructed Si(557) surface, where a triple step is combined with a single Si(111)7×7 unit. The driving forces for establishing regular step patterns are discussed. © *2001 American Institute of Physics.*
[DOI: 10.1063/1.1401788]

Experimental silicon devices are shrinking to dimensions of less than 50 nm. They call for fabrication methods beyond conventional lithography, such as extended ultraviolet (EUV) and x-ray lithography, or patterning via electron, atom, and ion beams. A frequent test pattern is a grating consisting of parallel, equispaced grooves.[1–3] Gratings have also been considered for nanometer metrology,[1,2] which plays an essential role in achieving proper overlay in lithography. In the single-digit nanometer regime, self-assembly methods become an attractive alternative to lithography. However, self-assembly is fraught with uncontrolled size distributions. The best of both worlds could be combined by a self-assembly process that produces structures with a well-defined number of atomic rows per period. That would be analogous to the self-assembly of atomically perfect molecules, such as the fullerenes.

We find that the Si(557) surface forms an atomically perfect grating structure with exactly 17 atomic rows of the bulk lattice defining the period of 5.73 nm. It is referenced directly to the lattice constant of Si, which has been used as a length standard.[4] The highly corrugated triple step structure of this surface is easier to detect than other long-range surface reconstructions and better suited for surviving exposure to air or liquids. These characteristics make the Si(557) surface a promising template for subsequent assembly of inorganic and organic nanostructures.[5] They might also find use as an atomically precise length reference in nanolithography.[1,2]

Previous nanoscale gratings span a wide range of periods, starting with high-precision 213 nm gratings obtained by optically driven atom deposition[1] down to 38 nm period patterns obtained by interferometric EUV lithography.[3] Self-assembled gratings with periods of about 80 nm have been obtained with faceted Si and GaAs surfaces.[6,7] They are far from being atomically precise, both in height and periodicity. On an even finer scale the natural reconstructions of high-index Si surfaces have been explored.[8] Structures with stable

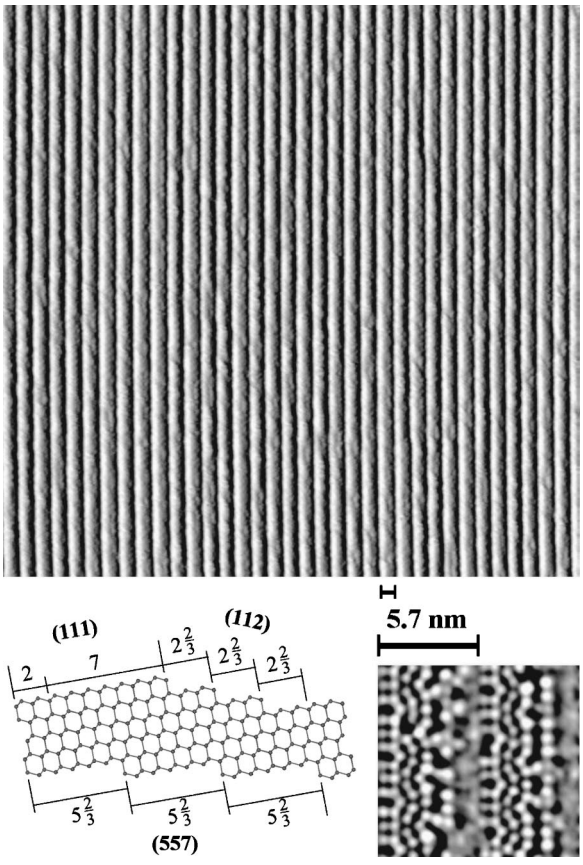

FIG. 1. STM images of a Si(557) surface with a periodicity of 5.73 nm, together with a side view of the unreconstructed unit cell. A single Si(111)7×7 unit is combined with a triple step that can be approximated by a Si(112) facet. The overview gives the *x* derivative of the topography. The closeup is high-pass filtered to show the surface atoms.

a)Author to whom correspondence should be addressed; electronic mail: fhimpsel@facstaff.wisc.edu

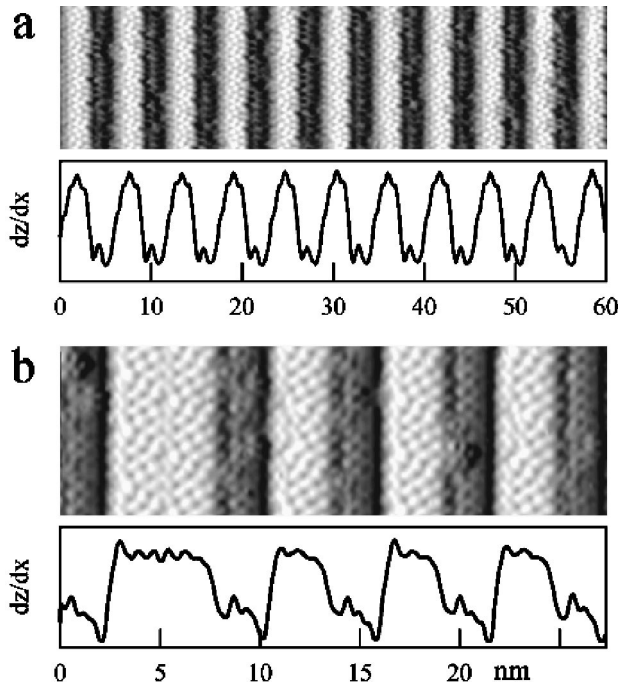

FIG. 2. STM images of the x derivative of the topography, combined with cross-sectional profiles averaged over the y axis. (a) A series of steps with the perfect periodicity of 17 atomic rows for Si(557). (b) A series containing a doubled Si(111)7×7 facet, which is used for calibrating the grating period.

facets have been identified, such as Si(112) and Si(5 5 12). Step structures with particularly smooth edges have been fabricated on vicinal Si(111)7×7 surfaces with a miscut toward the $[\bar{1}\,\bar{1}2]$ azimuth,[6,9] where the large 7×7 unit cell suppresses the formation of kinks. The density of kinks can be as low as one in 20 000 lattice sites.[9] Nevertheless, the step spacing of all these surfaces fluctuates.

We have set out to improve the regularity of step arrays and to push it all the way to the atomic limit, where the period is determined by an integer number of atomic rows. Thereby we follow the idea that reduced step spacing increases the step–step interaction. Repulsion between steps equalizes their distance. Elastic interactions between steps can be generalized according to their symmetry into monopole and dipole character. The monopole term originates from an elastic interaction between walls of strain domains.[10] It has a logarithmic dependence on step separation l. The elastic dipole interaction between steps is repulsive and has l^{-2} character.[11] The distribution of terrace widths on vicinal Si(111), as well as their fluctuations, are explained well by the dipole interaction.[12]

When the step spacing is reduced, triple steps partially replace single steps and create a nonperiodic structure.[13] We find that the triple steps take over completely when further increasing the miscut. A well-defined limit is reached when they are spaced by a single 7×7 unit. This surface has a 9.45° miscut angle and corresponds to Si(557), as shown in Fig. 1 in a side view. The bottom part of this side view shows the primitive Si(557) unit cell, the top part the actual unit cell, which is seven times larger parallel to the steps and three times larger perpendicular to them. Its dimensions of $2.69\times5.73=15.4$ nm^2 are substantial (compare $0.77\times5.35=4.1$ nm^2 for Si(5 5 12), one of the largest Si unit cells studied to date).[8]

Vicinal Si(111) surfaces with a miscut toward the $[\bar{1}\bar{1}2]$ azimuth can be viewed as a staircase of (111) terraces. The step edges are parallel to the $[\bar{1}10]$ direction. Triple steps have the special property of preserving the registry of the Si atoms on adjacent terraces, due to the abc stacking of the fcc lattice that underlies the diamond structure of Si. Surfaces with triple steps have the crystallographic notation $(n\,n\,n+6)$. The staircase has $N=n+2$ atomic rows per step. The row spacing is $d_{\mathrm{row}}=(3/8)^{1/2}\,a=0.3326$ nm, with the cubic lattice constant $a=0.543\,102\,088(16)$ nm for Si at 22.5 °C.[4] The period of the step lattice in the surface plane is $p=[N^2+8]^{1/2}d_{\mathrm{row}}$. The miscut angle from (111) is $\vartheta=\arctan\sqrt{8}/N$. For $n=15$ one obtains the (15 15 21) =(5 5 7) surface with $N=17$ atomic rows per (111) terrace, a period $p=5.73$ nm, and a miscut angle $\vartheta=9.45°$.

Strips of n-type, 1 Ω cm Si were heated by a current parallel to the steps. A 5 s flash to 1250 °C was followed by a ramp down to 1060 °C during 30 s, a 2 s quench to 830 °C, and a 15 min postanneal at 830 °C with slow cooldown.

Figure 1 (top) gives an overview of the long-range grating structure that can be achieved on Si(557). The closeup image of two periods (bottom) reveals a complex surface reconstruction. The side view decomposes Si(557) into a (111) facet with nine atomic rows and a (112) facet with eight rows, giving a total of 17 rows in the (111) projection. The actual (111) facet exhibits a 7×7 adatom pattern plus a string of Si adatoms that takes up two extra rows. The structure of the step facet is still under study.[14]

For calibrating the period of the Si(557) surface we use the Si(111)7×7 lattice that is part of the reconstruction [Figs. 2(a) and 2(b)]. Since the edges of a single 7×7 cell might be shifted by strain we use an anomalous (111) facet containing two 7×7 cells [Fig. 2(b)]. The period is measured by two independent methods: (1) A 7×7 grid is matched to the two 7×7 cells in the scanning tunneling microscope (STM) image and then extended over the rest of the (557) surface. (2) The widths of the wide cell and the normal (557) cell, p_{wide} and p, are determined from line profiles that are averaged in the y direction. The difference p_{wide}-p corresponds to seven atomic rows in the (111) plane. In the experiment the row spacing is projected onto the (557) plane by the z motion of the STM tip. As a consequence, the 7×7 triangles are not quite equilateral but contracted perpendicular to the steps by a factor $\cos\vartheta=0.986$. The 7×7 period that we measure is $p_{7\times7}=7d_{\mathrm{row}}\cos\vartheta=2.30$ nm, resulting in a (557) period $p=7d_{\mathrm{row}}\cos\vartheta\,p/p_{7\times7}$. The number of atomic rows in the (111) projection is $N=p\cos\vartheta/d_{\mathrm{row}}=7\cos^2\vartheta\,p/p_{7\times7}$.

The short-range periodicity can be measured reproducibly within 0.03 nm (0.1 atom spacing) by averaging over a set of line scans, such as in Fig. 2. Point defects due to adsorbates or displaced Si atoms are averaged out. The long-range periodicity of Si(557) is limited by three types of linear defects. The most frequent is a unit cell with 19 atomic rows, where the (112) facet is extended by two rows. The second defect is shown in Fig. 2(b), where the terrace on the left contains two 7×7 units instead of one. The third defect combines two triple steps. Figure 1 demonstrates that the second and third defects can be avoided for at least 35 periods (≈600 atomic rows). The 19 row defect can be avoided for at least ten periods (≈170 atomic rows), as shown in Fig.

2(a). It is suppressed by a fast quench through the $1\times1\rightarrow7\times7$ transition at 870 °C.

In summary, we demonstrate an atomically precise grating consisting of an array of triple steps on silicon with a spacing of 5.73 nm (17 atomic rows). The triple steps provide a strong surface corrugation, which increases their interaction and thereby equalizes their spacing. For future practical use it will be necessary to passivate the broken bonds of the clean Si surface, such that the structure survives in air. We have demonstrated two possible passivation methods: one using methanol, the other a gold coating with a titanium wetting layer.[15]

This work was supported by the NSF under Award Nos. DMR-9815416 and DMR-0079983.

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