# Regular step arrays on silicon

J. Viernow,[a] J.-L. Lin, D. Y. Petrovykh, F. M. Leibsle,[b] F. K. Men,[c] and F. J. Himpsel[d]
*Department of Physics, University of Wisconsin–Madison, Madison, Wisconsin 53706-1390*

(Received 4 November 1997; accepted for publication 23 December 1997)

Highly regular arrays of steps are produced on vicinal Si(111)7×7. The step edges are atomically straight for up to $2\times10^4$ lattice sites. The terraces are single domain, which produces a minimum kink width of 2.3 nm (half a 7×7 unit cell) and thus a high barrier for creating kinks. Criteria for obtaining optimum step arrays are established, such as the miscut [≈1° towards (112)] and an annealing sequence which passes through step bunching regions quickly. © *1998 American Institute of Physics.* [S0003-6951(98)03908-4]

The patterning of semiconductor surfaces on the nanometer scale has become important for a variety of applications, such as quantum wires and dots, as well as magnetoresistive sensors on sawtooth-shaped semiconductors.[1] Particularly appealing are self-assembly methods.[2–5] They are easier and faster than high-resolution lithography and writing, but they also tend to produce less-perfect structures. Therefore, obtaining straight edges and narrow-size distributions will be the key to practical use of self-assembled structures.

Stepped surfaces can serve as templates for producing one-dimensional wires or stripes by decorating the step edges in a step-flow growth mode.[2,3,5] For such structures it is essential to start out with smooth steps. Silicon, of course, is among the preferred substrates. There have been extensive studies of steps and kinks on silicon, including their kinetics and dynamics.[6–16] Our goal is to utilize the lessons from this work to optimize silicon substrates for step-flow growth of quantum wires. In particular, we want to avoid defects, such as kinks, step bunches, and multiple domains.

We are able to produce highly regular step arrays on Si(111)7×7, which exhibit atomically straight step edges. Kink densities as low as one in $2\times10^4$ lattice sites are achieved. The 7×7 reconstruction helps ordering the steps by quantizing the kink width in units of half the 7×7 unit cell (2.3 nm), which creates a large energetic and kinetic barrier for forming kinks. Several parameters have to be considered for perfecting the step arrays, such as miscut angle (azimuthal and polar), annealing sequence, current direction, and external stress.

Our choice of Si(111)7×7 as the substrate is motivated by the existence of a particularly stable step geometry on this surface. The top edge of an equilibrated step consists of a string of corner holes of the 7×7 unit cell,[6–10] which runs along the [110] direction. This still leaves two possible orientations of the step edge, where the surface normal is tilted from (111) towards opposite directions, i.e., (112) and (112). The steps with the (112) tilt are taken as the most stable configuration since they are found to occur during Si-on-Si(111) epitaxy.[7] The Si(100)2×1 surface has a more complex step structure consisting of alternating rough and smooth steps, which do not easily combine into smooth double steps.

We have studied Si(111) wafers with miscuts between 1° and 4° along (112) and (112). The doping was *n* type in the $10^{18}$ $cm^{-3}$ range. They were held as stress free as possible between Ta wire loops and heated by a dc current parallel to the direction of the step edges. The temperature was determined by a Minolta-Land Cyclops 52 optical pyrometer with the emissivity set to 0.4. The most regular step arrays were obtained from samples cut 1.1° towards (112) using a multistep annealing sequence. The wafers were outgassed for 1/2 h by raising the temperature slowly up to 700 °C, flashed to 1260 °C for 10 s to diffuse residual surface C into the bulk, and cooled to 1060 °C in 1 min, where single steps are stable. The most critical part is a quench to 850 °C within 3 s for avoiding a step tripling regime, and a 1/2 h postanneal at 850 °C and lower to develop long-range 7×7 domains. Scanning tunneling microscope (STM) images were taken at a tunneling current of 0.4 nA and a sample bias of +2 V. Step locations and terrace widths were determined by a computerized pattern recognition routine,[17] which allowed us to obtain quantitative step distributions from a large data set.

Figure 1 shows STM images from various step arrays on a Si(111) wafer, miscut by 1.1° towards the (112) direction. To enhance the steps, the derivative of the topography is presented. This can be viewed as the surface illuminated from the left side with the steps going downhill to the right. All steps are a double-layer high (0.31 nm), and the average terrace width of 15 nm corresponds closely to the nominal miscut of 1.1°. In the top panel one observes atomically straight step sections, interrupted by a series of kinks. All kinks have an identical width[18] of 2.3 nm. They all point to the right. These kinks are created by a small miscut in the azimuthal direction (≈1° away from the [112] direction). To determine the limit of spontaneously created kinks we have searched for spots on the Si wafer with the correct azimuthal orientation, such as on the bottom of Fig. 1. A lone kink is observed in this image over an area that comprises $2\times10^4$ step edge sites.[18] This kink density is significantly lower than the upper limit of 1 in $10^2$ sites set for H-terminated Si(111)

[a]Permanent address: Institut für Festkörperphysik, Universität Hannover, D-30167 Hannover, Germany.
[b]Permanent address: Department of Physics, University of Missouri Kansas City, Kansas City, MO 64110.
[c]Permanent address: Department of Physics, National Chung Cheng University, Taiwan, R.O.C.
[d]Electronic mail: himpsel@comb.physics.wisc.edu

 

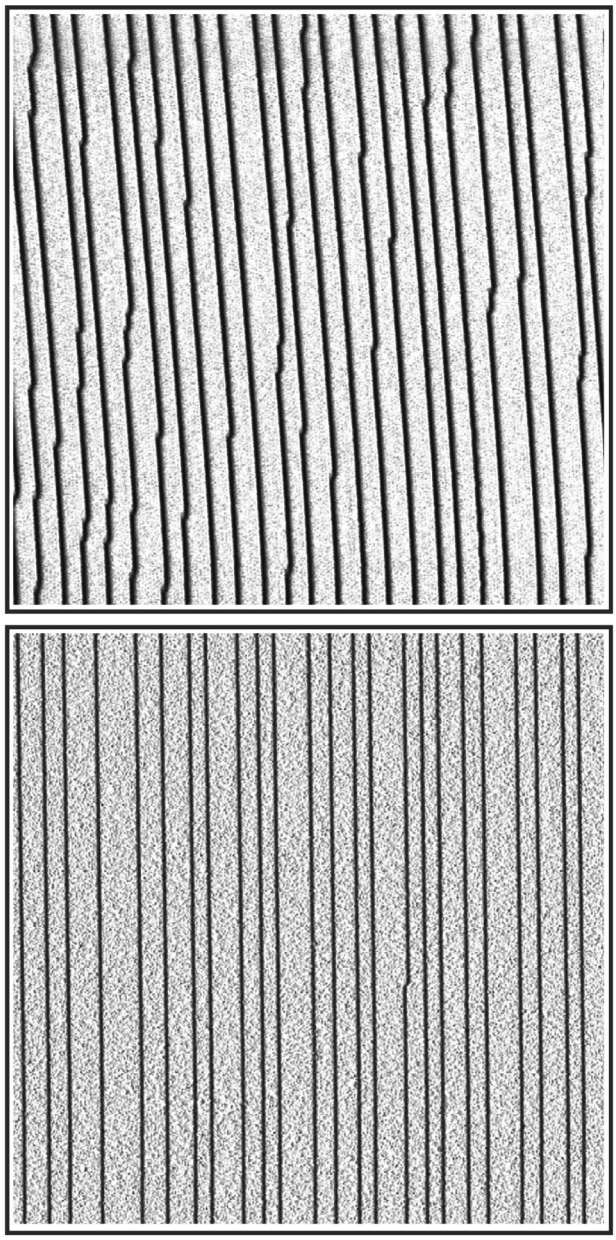

FIG. 1. STM images of step arrays obtained on Si(111)7×7 with a miscut of 1.1° towards the (112) direction (340×340 $nm^2$). The $x$ derivative of the topography is displayed to enhance steps (dark lines). In the top panel, an azimuthal miscut of ≈1° produces kinks, all having the same width (2.3 nm). The bottom panel contains only one kink in $2\times10^4$ step edge sites.

surfaces.[16] The steps on Si(111)7×7 are much smoother than those obtained on metal surfaces.[5]

The origin of such a low kink density lies in the 7×7 reconstruction of the surface. The upper step edge always runs along corner holes of the 7×7 unit cells[6–10] (see Fig. 2). A kink has to be at least one row spacing wide (2.3 nm), as long as the 7×7 reconstruction on a given terrace is single domain. This large size of a minimum kink provides a high energetic and kinetic barrier for kink creation[10,13] and explains the smoothness of the step edges observed on Si(111)7×7. In the following, we will quantify the perfection of the step arrays produced on Si(111)7×7 and give a list of criteria that optimize their growth.

For evaluating the order in step arrays we distinguish between the directions parallel and perpendicular to the step

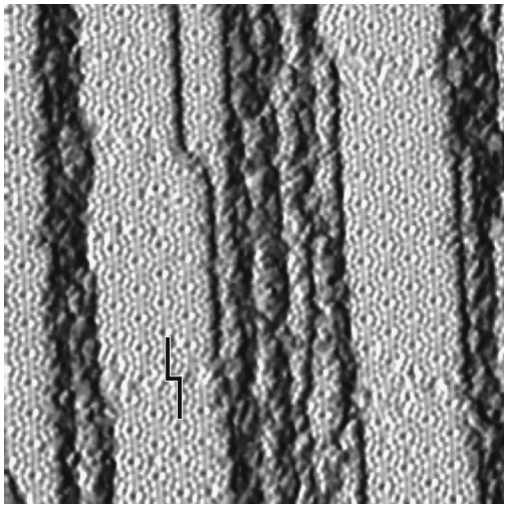

FIG. 2. STM image of an irregular step array obtained on Si(111)7×7 with a miscut of 3.5° towards the (112) direction (50 nm wide). Three types of characteristic defects are seen, i.e., step bunches, kinks, and domain boundaries (jog in the black line).

edges. The highest perfection is found parallel to the step edges along the [110] direction. Kink densities as low as 1 in $2\times10^4$ atom spacings (≈8 μm) can be achieved along that direction (Fig. 1, bottom). The quantized nature of the kink width implies that the 7×7 reconstruction remains single domain on each terrace over such distances. In fact, we have never observed a domain boundary for samples with optimum miscut and preparation. Domain boundaries do become visible in Fig. 2 for a sample miscut by an angle of 3.5°, which is too large and towards the wrong direction, i.e., (112). As indicated by the black line in Fig. 2, corner holes jog by a fraction of a 7×7 quantum across a domain boundary, making kinks with fractional width possible.

Going from one terrace to the next, we find the order to be less perfect. As observed previously,[13,14] several of the possible 7×7=49 registrations do indeed occur between the

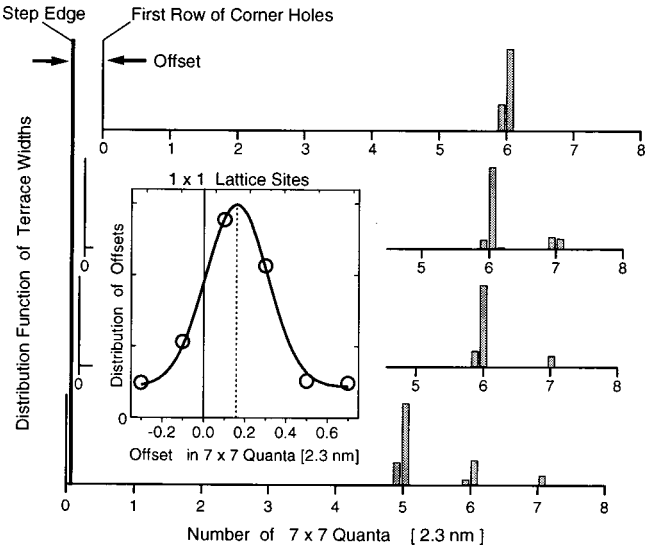


FIG. 3. Width distributions of individual terraces in Fig. 1 (histograms). They are characterized by a series of δ functions, separated by half a 7×7 unit cell (2.3 nm). When averaged over all terraces, the δ functions become convoluted by the distribution of offsets between a step and the first row of corner holes below it (see the inset).

7×7 domains on adjacent terraces. Among the seven registrations parallel to the step edge, we find a prevalence of those who preserve the (110) mirror plane symmetry. The rows of corner holes perpendicular to the step edges continue from one terrace to the next in this case (not shown). Perpendicular to the step edge, the seven possible registrations induce different offsets between the step edge and the first row of corner holes on the lower terrace (Fig. 3). The most commonly observed offset is about 0.2 of a 7×7 quantum (≈0.5 nm or one 1×1 lattice site). This offset affects the distribution of terrace widths in Fig. 3. The most commonly observed offset is about 0.2 of a 7×7 quantum (≈0.5 nm or one 1×1 lattice site). This offset affects the distribution of terrace widths in Fig. 3. For a given terrace we find a series of $\delta$ functions (within our pixelation limit), with an increment of one 7×7 quantum generated by a kink. These $\delta$ functions are shifted slightly from one terrace to the next, due to the variations in the offset at the step edge. The overall terrace width distribution exhibits a standard deviation $\sigma$ of 3–4 nm (≈1–2 quanta), which is comparable to the value of $\sigma$=3.6 nm (1.6 quantum) obtained for samples with similar miscut previously.[10]

In producing highly regular step arrays on silicon, we had to overcome a variety of obstacles, which are illustrated in Fig. 2. Irregular or bunched steps may occur. Multiple 7×7 domains may form on a terrace and give rise to kinks narrower than the 7×7 quantum (see the jog in the rows of 7×7 corner holes indicated by a black line in Fig. 2). An important part of our study is the systematic minimization of such detrimental effects. The following parameters are found to be relevant in this process:

(1) Miscut angle, polar, and azimuthal: Step bunches or triple steps[9–11] are difficult to avoid for polar miscut angles larger than about 2°. Diffusion across shorter terraces takes less time and facilitates the formation of multiple steps. Triple steps are formed just below the 7×7-to-1×1 phase transition at 870 °C on miscuts towards (112), step bunches (facets) on miscuts towards (112).
(2) Annealing temperature and time: Our strategy in developing the optimum annealing sequence is to quickly quench the wafer through temperature regions where step bunching or tripling occurs. Thermal disorder from the quench is removed in a postanneal at lower temperatures, where the mobility is too low for step bunching. The most critical part is the temperature of 850 °C for the postanneal. If this temperature is too high, i.e., too close to the 7×7-to-1×1 phase transition at 870 °C, some of the single steps convert to triple steps.[10] If it is too low, thermal disorder from the initial flashing is not annealed out completely.
(3) Current direction: The heating current is passed through the wafer parallel to the step edges to avoid step bunching due to electromigration.[15] By symmetry, this effect can only happen in the perpendicular geometry.
(4) Stress: We minimize stress by holding the wafer between soft Ta wire loops where it can expand freely upon heating. Stress-induced step bunching is well documented[19] on Si(100).

In summary, we are able to produce arrays with unprecedented perfection on Si(111) by a proper choice of the wafer miscut and by an annealing sequence that passes quickly through step bunching regions. The kink density is extremely low, making Si(111)7×7 steps ideal for creating quantum wires[2,3,5,20] by growth along atomically straight steps. We are in the process of fabricating such structures, e.g., epitaxial $CaF_2$(111) stripes[20] that can play the role of a photoresist in traditional microlithography.

This work was supported by NSF under Award Nos. DMR-9624753 and DMR-9632527.

[1] T. Ono and T. Shinjo, J. Phys. Soc. Jpn. **64**, 363 (1995); M. A. M. Gijs, M. Johnson, A. Reinders, P. Huisman, R. van de Veerdonk, S. Lenczowski, and R. van Gansewinkel, Appl. Phys. Lett. **66**, 1839 (1995); W. Oepts *et al.*, Phys. Rev. B **53**, 14 024 (1996).
[2] P. M. Petroff, A. C. Gossard, and W. Wiegmann, Appl. Phys. Lett. **45**, 620 (1984); M. Tsuchiya, P. M. Petroff, and L. A. Coldren *ibid.* **54**, 1690 (1989).
[3] R. Nötzel, N. N. Lendentsov, L. Däweritz, K. Ploog, and M. Hohenstein, Phys. Rev. B **45**, 3507 (1992).
[4] Y. H. Phang, C. Teichert, M. G. Lagally, L. J. Peticolos, J. C. Bean, and E. Kasper, Phys. Rev. B **50**, 14 435 (1994); J. Tersoff, C. Teichert, and M. G. Lagally, Phys. Rev. Lett. **76**, 1675 (1996).
[5] F. J. Himpsel, Y. W. Mo, T. J. Jung, J. E. Ortega, G. J. Mankey, and R. F. Willis, Superlattices Microstruct. **15**, 237 (1994); T. Jung, R. Schlitter, J. K. Gimzewski, and F. J. Himpsel, Appl. Phys. A **61**, 467 (1995).
[6] R. S. Becker, J. A. Golovchenko, E. G. McRae, and B. S. Swartzentruber, Phys. Rev. Lett. **55**, 2028 (1985).
[7] R. J. Hamers, U. M. Köhler, and J. E. Demuth, Ultramicroscopy **31**, 10 (1989).
[8] R. Wiesendanger, G. Tarrach, D. Bürgler, and H.-J. Güntherodt, Europhys. Lett. **12**, 57 (1990).
[9] R. J. Phaneuf and E. D. Williams, Phys. Rev. B **41**, 2991 (1990).
[10] E. D. Williams and N. C. Bartelt, Science **251**, 393 (1991), and references therein.
[11] M. Hanbücken, B. Röttger, R. Kliese, I. Vianey, and H. Neddermeyer, Europhys. Lett. **23**, 573 (1993).
[12] J. D. O'Mahony, J. F. McGilp, F. M. Leibsle, P. Weightman, and C. F. Flipse, Semicond. Sci. Technol. **8**, 495 (1993).
[13] J. L. Goldberg, X.-S. Wang, J. Wei, N. C. Bartelt, and E. D. Williams, J. Vac. Sci. Technol. A **9**, 1868 (1991).
[14] Y. Wang and T. T. Tsong, Phys. Rev. B **53**, 6915 (1996).
[15] Y.-N. Yang, E. S. Fu, and E. D. Williams, Surf. Sci. **356**, 101 (1996), and references therein.
[16] P. Jakob, Y. J. Chabal, K. Kuhnke, and S. B. Christman, Surf. Sci. **302**, 49 (1993).
[17] D. Y. Petrovykh, F. J. Himpsel, and T. Jung (unpublished).
[18] The quantum for a kink width is half of the long diagonal of the 7×7 unit cell, i.e., a $(7\sqrt{6})/4=2.3$ nm with the cubic lattice constant $a=0.54$ nm. The spacing between sites along the [110] step direction is $a/\sqrt{2}=0.38$ nm.
[19] M. B. Webb, Surf. Sci. **299/300**, 454 (1994).
[20] J. Viernow, J. Lin, D. Petrovykh, F. Men, D. Seo, M. Henzler, and F. J. Himpsel (unpublished).