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Simple Si(111) surface preparation by thin wafer cleavage

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We describe a simple method to obtain clean Si(111)2×1 surfaces with large terrace sizes, up to tens of microns, using commonly available wafers. The sample geometry and cleavage method make it possible to produce semiconductor samples in ultra-high vacuum systems: that are not explicitly designed to cleave samples or prepare semiconductor surfaces by other means. The force required to cleave the samples is sufficiently low such that they can be cleaved using any available transfer arm, manipulator, or wobble stick in the vacuum system. Large atomic terraces on the order of hundreds of nanometers to tens of microns are easily obtained in this way, as characterized by scanning tunneling microscopy. © *2013 American Vacuum Society*. [http://dx.doi.org/10.1116/1.4790475]

I. INTRODUCTION

Silicon is a very common and useful material for surface science studies, but its reliable preparation requires careful mounting to a sample holder, delicate control over flash annealing parameters, challenging thermometry, and extreme care in specimen handling in order to avoid contamination.[1] We describe a procedure for the preparation of high quality Si(111)2×1 surfaces by cleavage of a thin silicon wafer. The technique is suitable for straightforward implementation in ultra-high vacuum surface science systems that are not explicitly designed to prepare semiconductor samples by either flash annealing or cleaving methods. The ease of sample preparation also makes these samples ideal starting points for the studies with the Si(111)-5×5 and 7×7 surfaces by subsequent annealing.[2,3]

During a study on the interaction of atomically defined field ion microscope tips in scanning tunneling microscopy (STM), we required substrates that were much more chemically reactive than the standard metal surfaces we prepare routinely.[4] As our ultra-high vacuum (UHV) system's surface preparation tools are oriented toward metal surfaces (ion sputtering and resistive annealing), it was impossible to prepare silicon surfaces for study in STM without performing substantial system renovations. This initiated our investigation into the preparation of silicon surfaces by cleavage.

Cleavage in UHV has been used to prepare many types of crystal surfaces with appropriate quality for scanning probe studies. The (100) faces of rocksalt crystals such as KBr, NaCl, and NiO can be cleaved with a special anvil or blade apparatus actuated from a vacuum feedthrough.[5,6] Layered oxides can be cleaved but often present challenges since the surface quality degrades at elevated temperatures, requiring the cleavage apparatus to be cooled to cryogenic temperatures.[7,8] Closely related to our type of sample preparation is the cleavage of (110) faces of III-V semiconductors, studied extensively in cross-sectional STM.[9–13] Since the (110) face is perpendicular to the surface of (100) oriented wafers, cleavage is more easily carried out than for our Si samples. Si(111) surfaces have previously been prepared using various cleavage methods resulting in different surface qualities.[14–18] We report in detail a reliable method to expose Si(111) surfaces with large terraces and show how cleavage can be accomplished without special system modifications other than the sample holder for the wafer piece.

Due to the simplicity and reproducibility of preparing the cleaved Si(111)2×1 samples, we consider them very suitable for testing STM functionality, a procedure often reserved for highly oriented pyrolytic graphite (HOPG). Two notable advantages for using Si(111)2×1 samples over HOPG suggest themselves. First, true atomic resolution can be verified, as opposed to lattice resolution obtained on HOPG by scanning commensurate carbon flakes over each other.[19,20] Second, the tunneling gap is exceptionally stable and free of spikes resulting from the transfer of physisorbed species or carbon flakes. The stable tunneling conditions can be used as a sensitive monitor of tip-sample vibrations: after obtaining a current versus distance curve to characterize the tunneling barrier, variations in the tunneling current recorded in the absence of distance feedback can be related to tip-sample distance modulations. In such a manner, we successfully diagnosed (and fixed) a source of line frequency (60 Hz) tip-sample vibrations coming from our coarse approach motor electronics.

Since the UHV system lacked a formal sample cleaving assembly, we began experimenting with different methods of mounting silicon wafer samples so that they could be cleaved using regular UHV manipulator arms. This required a sample that could be cleaved with a relatively low force to ensure that the manipulators were not damaged. In addition, the surface had to be homogeneously flat so that a STM tip could be coarse approached by monitoring its reflection in the sample. These requirements influenced our choice of wafer orientation, sample size, and method of scribing to

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initiate cleavage. The result of our successively refined sample preparation procedure is described below.

II. SAMPLE PREPARATION

We start with a 0.26 mm thick, 2 in. diameter Si(111) wafer (Boron doped, 10–15 Ω cm), polished on the top side. Si(111) is chosen over Si(100) because the (111) cleavage plane is closer to perpendicular to the wafer's surface ($\theta_{(111)-(11\bar{1})} \simeq 70.5^\circ$ whereas $\theta_{(100)-(111)} \simeq 54.7^\circ$). We use a thin 0.26 mm wafer to reduce the force necessary to cleave the sample. The use of a Si(110) wafer would allow for a cleaved surface perpendicular to the wafer; however, this crystal orientation is very difficult to find in thin wafers.

The Si(111) wafer is first sectioned into strips parallel to the $(1\bar{1}0)$ primary flat with a width of ~3 mm, as illustrated in Fig. 1(a). These are cleaved by scoring the sample with a diamond scribe and partitioning them over the edge of a glass slide. The sample strips are then sectioned into ~12 mm pieces, shown in Fig. 1(b), by the same diamond scribing and cleaving process. Finally, a small scratch to initiate the cleavage is scored on the top (polished) side of the wafer using the diamond scribe. The scratch extends from the edge of the sample to a length of ~1 mm, illustrated by the white line in Fig. 1(b). KOH can also be used to create a notch in the wafer to initiate cleavage,[18] but we have found scribed scratches to produce cleaved surfaces of sufficient quality. Some care should be taken to ensure the scratch is straight—use of a glass slide as a straight-edge is advisable over free-hand scribing.

A force applied as shown in Fig. 1(c) will cleave the sample to expose the $(11\bar{1})$ plane as indicated. A force of only 0.25–0.35 N (25–35 g) is required to initiate cleavage, as measured on a digital scale. The required force is sufficiently low that it can be supplied by any convenient transfer arm or wobble stick in the UHV system, eliminating the need for a dedicated sample cleaving apparatus.

A photograph of the mounted sample is shown in Fig. 2(a) along with the direction of the applied force. The sample is mounted on a custom sample holder designed to hold it at the appropriate 70.5° angle such that the cleavage plane is parallel

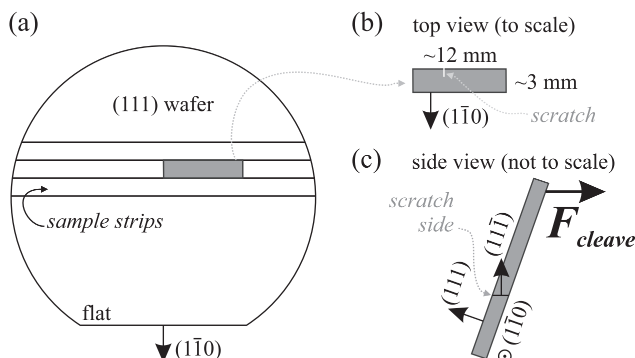

FIG. 1. (a) Two inch diameter Si(111) wafer is sectioned into sample strips. The sample strips are further sectioned smaller pieces, shown in (b). (b) Top view of the ~12 × 3 mm sample pieces. The scratch used to initiate cleavage is shown as a white line. (c) Side view, where the orientation of the $(1\bar{1}0)$ wafer flat points out of the page, and the $(11\bar{1})$ surface to be exposed by cleavage points upwards. The location of the scratch and direction of the cleavage force are indicated.

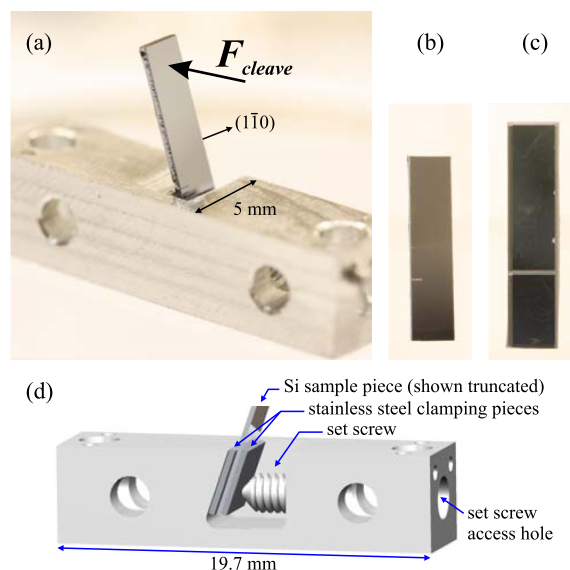

FIG. 2. (Color online) (a) Sample holder and mounted Si sample piece. (b) Si(111) sample piece with small scribed scratch to initiate cleavage. (c) Less successful Si(100) sample piece cut half-way through with diamond saw in an attempt to reduce cleavage force (see text). (d) CAD rendering of the sample holder showing details of the clamping system.

to its top surface. The prepared sample, with the small diamond scribe scratch, is shown in Fig. 2(b) prior to mounting in the holder. The clamping system of the wafer piece is illustrated by a CAD rendering shown in Fig. 2(d). The Si sample piece is clamped between two $3.5 \times 3.5 \times 0.3$ mm^3 pieces of 430 stainless steel sheet metal. These clamping pieces help to prevent the brittle fracture of the sample when clamped by the set screw. An 18-8 stainless steel M2 conical point set screw provides the clamping force and a hole at the end of the sample holder allows access to the screw head.

Initially, we investigated the possibility of using thicker wafers that were diced half-way through their thickness to reduce the force necessary for cleavage, as shown in Fig. 2(c). Although the dicing was successful in reducing the cleavage force and in defining the location at which cleavage would occur, the surface quality was very poor because of the multitude of crack initiation points. The surfaces showed very small terraces, rarely exceeding several nanometers, and were not macroscopically smooth over large distances, making STM tip approach (aligned by optical reflection) very difficult.

After loading into the UHV system, the samples are degassed overnight at ~220 °C. The samples are cooled to room temperature before cleaving to minimize drift once transferred to the STM. In order to cleave off the top portion of the wafer, the sample holder is first secured to our sample preparation stage. Then, a sample transfer fork attached to a 3 axis manipulator is used to cleave the wafer by pressing on the top edge in the direction indicated in Fig. 2(a).

III. CHARACTERIZATION BY STM

The surface topography is macroscopically rough near the scribed scratch used to initiate cleavage. At the side opposite the scratch, the surface is exceptionally smooth. We have performed STM within 1 mm from the edge opposite the

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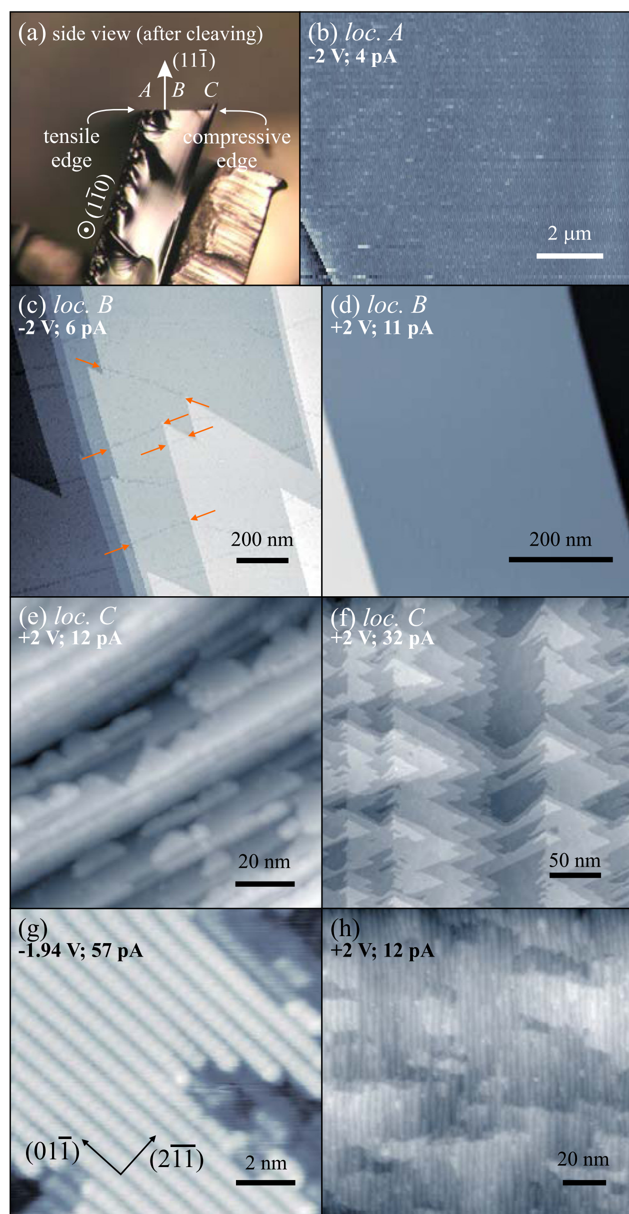

FIG. 3. (Color online) (a) Side view of sample after cleavage, in the same orientation as Fig. 1(c). STM scan locations A, B, and C relative to the tensile and compressive edges are indicated. (b) Extremely large atomically flat terrace at location A extending $> 10\,\mu$m, showing a single atomic step in the lower left corner. (c) and (d) Surface morphology near the center of the sample, location B. Arrows in (c) indicate antiphase boundaries of the reconstruction. (e) and (f) Surface morphology near the compressive edge, location C. (g) Atomic resolution of the 2×1 reconstruction. (h) Surface morphology of a more disordered region. STM imaging parameters indicate bias applied to the sample.

scratch with excellent reproducibility. The cleaved Si(111) has a width of $\sim$0.28 mm spanning from the tensile to the compressive edge of the sample (the force is applied by pushing on the tensile side). These edges are indicated in Fig. 3(a).

We now briefly describe surface topographies observed at locations spanning from the tensile to compressive edges, labeled as locations *A*, *B*, and *C* on the side view of the cleaved edge in Fig. 3(a). These are representative of topographic features observed in the half-dozen samples we have imaged by STM.

At location *A* near the tensile edge, extraordinarily large terraces are found, and can exceed $10\,\mu$m between atomic steps. Figure 3(b) presents a STM topograph showing a single atomic step in the lower left corner and a flat terrace exceeding $10\,\mu$m in size. The image has low resolution on the vertical axis in order to compensate for the long acquisition time needed for this scan size.

Near the center of the cleaved edge (location *B*), approximately equidistant between tensile and compressive edges, the atomic terraces show sizes on the order of hundreds of nanometers, as shown in Figs. 3(c) and 3(d). The terraces in Fig. 3(c) are separated by single atomic steps of height $\sim$0.3 nm. In Fig. 3(d), bunches of about ten steps separate atomically flat terraces. The terraces display the well-ordered 2×1 Pandey reconstruction[21] (not resolved at this scale), formed spontaneously upon cleavage. Antiphase boundaries between reconstructed domains are visible as darker lines, which extend across terraces, highlighted by arrows in Fig. 3(c). They are visible in this large scan frame because the domain boundaries are enlarged by rest gas molecules selectively binding to the more reactive sites at the edge of reconstructed domains. These stripes of adsorbed rest gases widen with time and eventually completely deplete the 2×1 reconstructed surface.

A rough estimation from sequential STM scans of the same area reveals that the coverage of adsorbed molecules (dark regions) expand from $\sim$10% to $\sim$28% over a delay of 1.5 h. Assuming that most of the UHV rest gas constituting the base pressure of $\sim 7 \times 10^{-11}$ mbar is H_2, we estimate a dose of $\sim$0.9 L during this time (corrected for ionization gauge sensitivity[22] for H_2) This estimation suggests a sticking coefficient $(0.28–0.10)/0.9 = 0.2$, which is somewhat less than unity (neglecting gauge calibration, rest gas composition, and surface binding site density).

At the compressive edge of the sample (location *C*), the surface generally shows much smaller terraces, often with small isolated single-layer high regions, shown in Figs. 3(e) and 3(f). There is a larger local variability in surface structure around location *C* than locations *A* or *B*, but the surface may still be very appropriate for some surface studies.

Resolution of the 2×1 surface reconstruction is shown in the constant current STM image of Fig. 3(g). A corrugation of $\sim$50 pm is measured in the $(2\bar{1}\bar{1})$ direction and $\sim$5 pm in the $(01\bar{1})$ direction at -1.94 V sample bias. Upon applying a positive sample bias of $+2$ V, the corrugation in the $(2\bar{1}\bar{1})$ direction is reduced somewhat to $\sim$40 pm (image not shown).

In a minority of locations of our STM studies, we have found areas that were not as well ordered as those described above. One such surface morphology is illustrated in Fig. 3(h), consisting of a high density of steps with lateral size of $\sim$3 nm and larger scale modulations of the stepped structure. Although this type of surface is less well-defined for surface science studies, STM scanning is very stable and safe—the disordered surface does not damage the integrity of the tip.

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IV. SUMMARY

To summarize, high quality Si(111)2×1 surfaces can be produced by cleavage of thin wafers. Using the method we describe, silicon surfaces can be easily prepared in UHV surface science systems that were not previously designed for preparation of semiconductor surfaces. The force required to initiate cleavage is suitably low such that it can be supplied by any convenient transfer arm in the UHV system. The cleaved samples show atomically flat terraces with lateral dimensions exceeding 10 μm near the tensile edge and on the order of hundreds on nanometers near the center of the sample.

ACKNOWLEDGMENTS

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