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Surface stress on Si(001)2×1 surfaces studied by TEM

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Abstract. Thin Si(001) films were studied by transmission electron microscopy (TEM). Arrays of single steps were observed as alternately arranged bright and dark lines. The image contrast was weak where the lines were parallel to an operating reflection vector. Strains of the Si lattice around the steps were calculated using surface stress on the 2×1 reconstructed surface and the results explained the observed image contrast semiquantitatively.

1. Introduction

Ultra-high vacuum transmission electron microscopy and diffraction (UHV-TEM–TED) has been successfully applied to studies of surface structures and surface dynamic processes [1–7]. In the case of Si(111), structure analysis of Si(111)7×7 [3], studies of steps and out of phase boundaries (OPBS) on Si(111)7×7 surfaces [6, 8, 9], high-resolution imaging of Si(111)7×7 [10, 11], Si(111)5×1–Au [11] and Si(111)$\sqrt{3}\times\sqrt{3}$–Bi [12] and observation of a phase transition between the 7×7 and the 1×1 structures [13] have been reported. In the case of Si(001), studies of single- and double-step configurations on vicinal surfaces [14] and high-resolution imaging [11] have been reported. TEM studies on other Si surfaces have also been carried out [4, 15].

The dimer structure of Si(001)2×1 is well known to give rise to anisotropy in various surface dynamic processes. Diffusion of adatoms [16] and vacancies [17], adsorption processes of alkali metals [18] and current effects on the surface step structures, hence on surface domain structures [19, 20] have been reported to be anisotropic. The dimer structure is quite stable and it remains on the surface even above 1100 °C, at which sublimation takes place with an appreciable speed and quick motion of steps due to sublimation was observed [21].

The dimerization produces surface stress on the 2×1 reconstructed surface as is easily seen from its structural model. The dimerization favours compressive substrate lattice strain parallel to the dimer bond. However, a rigid substrate lattice leads to the reconstructed surface being under a strong tensile stress [22]. Thus, uniaxial compression and dilatation of the substrate surface lattice along [110] favours one of the two domains on its surface, and this has been studied extensively by Madison's group using low-energy electron diffraction (LEED) [23] and scanning tunnelling microscopy

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Table 1. Stresses of Si(001)2×1 surfaces. For comparison stress on Si(111)7×7 surfaces is also shown (after [22]).

Parallel to dimer bond	1.56 eV/(1×1 unit cell)
Perpendicular to dimer bond	−0.88 eV/(1×1 unit cell)
Si(111)7×7	2.6 eV/(1×1 unit cell)

(STM) [24]. Surface stress on the 2×1 surface has been studied theoretically and surface stresses listed in table 1 have been reported [22].

An experimental technique sometimes used to study surface stress is to observe bending of a rectangular thin film, one short edge of which is clamped. This method, however, can detect surface stress only when there is change in the surface stress on one surface or different changes on both surfaces. Also it cannot be used for multi-domain surfaces. On the other hand TEM can see strains produced by surface stress if there are boundaries on the surface. To date, a tensile stress in pseudomorphic Pd monolayer islands on an Ag substrate [1] and that in the Si(111)7×7 structure have been observed [13, 25].

In the present paper TEM observations of single steps on surfaces of Si(001) thin films are described. The steps were observed as an array of alternating bright and dark lines. The image contrast of the steps was analysed in terms of surface stresses listed in table 1. An elastic interaction between a step structure and a sub-surface dislocation was observed.

2. Experimental

A UHV electron microscope equipped with a specimen heating holder and a gas inlet system [6, 8] was used. Specimens, $1 \times 10 \times 0.4$ mm^3 in size, were cut from B-doped Si(001) wafer (a few ohm centimetres). A technique previously developed to obtain thin Si films was used [6]. After mechanical grinding to form a thin part, 0.5 mm in diameter and about 10 μm in thickness, the specimens were cleaned chemically and were clamped between two electrodes of the specimen holder. By resistive heating around 1200 °C at the specimen position of the microscope, thin regions, a few tens of nanometres thick, transparent to 100 kV electrons, were formed. The specimen was then cooled to around 800 °C and oxygen gas at a level of 10^{-6} Pa was introduced. This is to etch the thin parts further by oxide formation and subsequent sublimation of oxide. This allows us to make thin parts 10–20 nm in thickness [6].

3. Results

Figure 1 reproduces a bright field (BF) image (*a*) and a (220) dark field (DF) image (*b*) of an Si(001) film. The film temperature was about 730 °C. A TED pattern (not shown here) from the film showed reflections from two domains of the 2×1 reconstruction. Bright and dark line images, indicated by b and d, respectively, in (*a*) and (*b*), are considered to be surface atomic steps. At this temperature sublimation of Si atoms from the surface was not negligible and the line images moved upward. Therefore, the steps in (*b*) taken several minutes after (*a*) are not at different positions from those in (*a*). Due to the movement, we could identify step-down directions and these are indicated by arrow

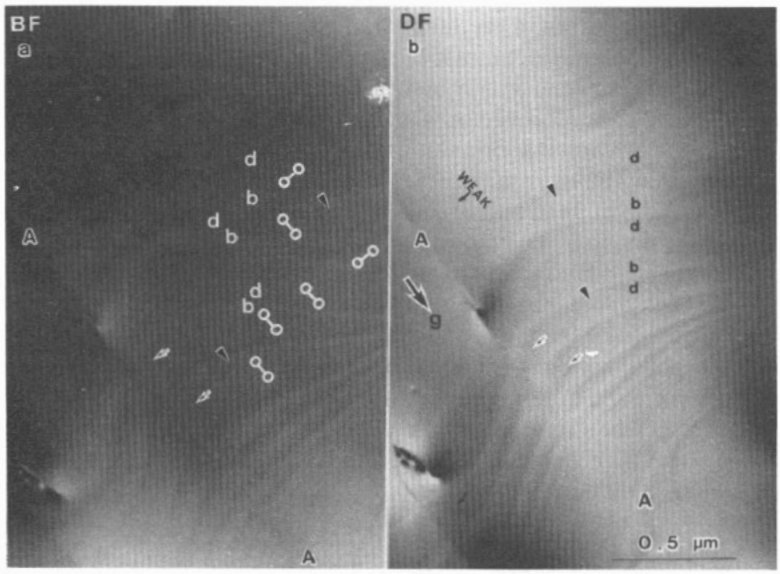

Figure 1. Bright and (220) dark field TEM images of an Si(001) film taken at 730 °C. Bright and dark line images are single steps on the 2×1 reconstructed surface. The step-down directions are indicated by arrow heads. Dimer marks, which were included to show that the steps are single steps, were not determined uniquely. An interaction of a dislocation along a line AA with the steps and surface structure domain terraces is seen (see the text for details).

heads in the figure; however, we could not tell whether the bright lines in (*a*) are dark or bright in (*b*). Also we could not discriminate whether the steps are on the top or bottom surfaces of the film as we could for steps on Si(111) [8].

There were several reasons why we considered the surface steps to be single steps whose step height is $\frac{1}{4}$ of the lattice constant of Si.

(1) The line images moved at high temperature, probably due to sublimation.

(2) An alteration of bright and dark lines was noted not only in the bright field image (*a*) but also in the dark field image (*b*). This alteration is consistent with that of single steps on (001)2×1 surfaces: steps with higher side terraces of the 2×1 domain and steps with higher side terraces of the 1×2 domain. To show the latter alteration one possible scheme for dimer directions is schematically shown in (*a*), although we did not determine the directions uniquely and directions perpendicular to those shown in (*a*) are also probable.

(3) The bright and dark images are due to lattice strain of the Si film caused by line forces at the single steps (see the discussion in section 4). The fact that the contrast was due to lattice strain with displacement vectors normal to the step line was deduced from the observation that the large image contrast of the steps was weak where the step lines were parallel to the reflection vector g (see a place indicated by 'WEAK' in (*b*) and a place marked C in figure 2).

It is considered that the steps exist on both surfaces of the film and may cross each other in the TEM images. In figure 1 there are no such steps, which indicated that one side surface was very flat. This was not always the case and we did observe crossing of steps in other images.

There is a dislocation in the film parallel to the surface along a line marked by AA in figure 1(*a*). The image contrast of the dislocation is rather weak under the present diffraction conditions and was found to be strong under the Bragg condition for the reflection whose reflection vector was perpendicular to the dislocation line. Thus, the dislocation is nearly in edge orientation. It is noted that the surface steps are curved at places where they run over the dislocation, indicated by arrows in (*a*) and (*b*). It is seen in (*a*) that pairing of dark and bright image steps, seen at the lower right-hand corner of the figure, comes apart as they approach the dislocation and interchanges of pairing occur there. Similar interaction of the steps with the dislocation is also seen in (*b*).

Two reasons are conceived for such interactions. One is an elastic interaction between lattice strain around the step and stress around the dislocation. When the step is exactly perpendicular to the edge dislocations, the interaction is expected to be weak. The other is an elastic interaction between the surface stress of the 2×1 structure and strain at the surface produced by the edge dislocation. In the present case the dimer bonds are parallel or perpendicular to the Burgers vector and the interaction favours one domain as is seen in figure 1(*a*).

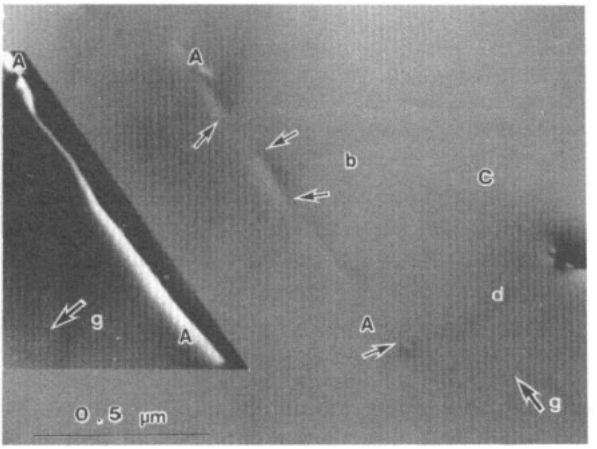

Figure 2. A (220) dark field image of an Si(001) film taken at 730 °C. A contrast reversal of a curved step from bright (b) to dark (d) and an interaction of a dislocation and the surface terraces are noted (see the text for details).

Figure 2 reproduces a 220 dark field TEM image of an Si(001) film taken at 730 °C. A single step marked by b is bright. The step is curved at C, where the image contrast is lost, and reappears as a dark line at a place marked by d. This fact again suggests that bright and dark line images are steps, because the contrast reversal is due to the reversal of the sense of strain resulting from the reversal of the step direction.

Another point to be noted is that segments of dark lines are seen along a direction indicated by AA and ends of the segments are at places where steps cross the lines. From the contrast analysis (see the dislocation with strong contrast in a dark inset in the left-hand side of figure 2, taken under the reflection vector perpendicular to the dislocation line) it was found that the segments are parts of one edge dislocation along AA. Since the reflection vector is perpendicular to the Burgers vector of the edge dislocation and the dislocation line, no contrast is expected. The observation of the contrast of the

dislocation with a strong correlation with the 2×1 terrace structure suggests a sort of elastic interaction between the dislocation and the surface strain of the 2×1 structure.

4. Discussion

It was found that the single steps on Si(111)2×1 surfaces were seen in TEM micrographs as alternating bright and dark lines because of lattice strain around the steps, and that there was elastic interaction between the edge dislocation in the film and the surface step structure.

From the theoretically estimated values of the surface stresses shown in table 1, strains of the Si crystal lattice were calculated using equations derived to calculate lattice strain along the edges of patches of Si oxide formed on Si [26]. Figure 3 reproduces our calculated results.

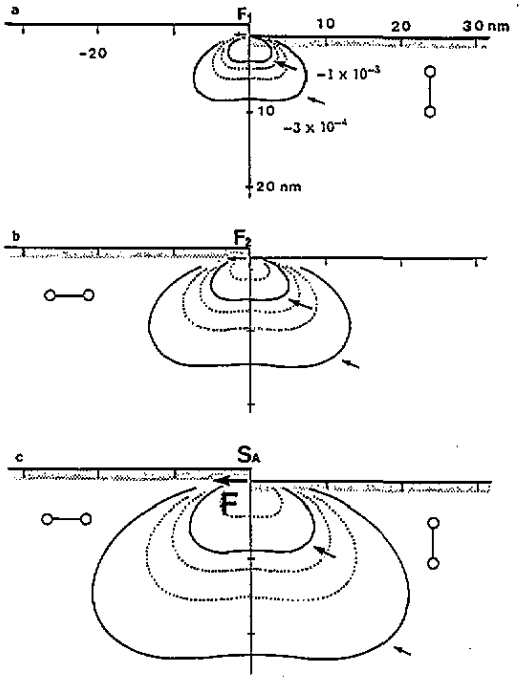

Figure 3. Calculated lattice strains (*c*) around a single step (S_A) on the 2×1 surface assuming surface stresses listed in table 1. Effects of the reconstruction on the lower and upper terraces are shown in (*a*) and (*b*) for comparison (see the text for details).

A single atom step of the S_A type was assumed as shown in (*c*): on the upper terrace the dimer bonds are perpendicular to the step and on the lower terrace they are parallel to the step. From table 1 it is seen that on the lower 1×2 terrace the surface is compressed along the dimer row direction (horizontal in the drawing) which causes a line force (F_1) to the left of the step as shown in (*a*). On the upper terrace, on the other hand, the surface stress is tensile and along the dimer bond (horizontal in the drawing) which causes a line force (F_2) to the left of the step as shown in (*b*). At the S_A step both forces act together at the step.

Calculated strains (tilt of vertical lattice planes) for each component of the force and the sum of them are reproduced in (*a*), (*b*) and (*c*), respectively, to show the relative weight of the two forces. The inner thick curves indicated by long arrows are for -1×10^{-3} and the outer thick curves indicated by smaller arrows are for -3×10^{-4}. It is seen that strained regions of the latter magnitude of strain, which can be easily detected by TEM, are as large as 40 nm in (*c*). It should be noted that widths of the step images in figures 1 and 2 are of similar values. For quantitative analyses, image widths taken under various diffraction conditions should be compared with calculated image contrast based upon the strain field shown in (*c*), and this is yet to be solved.

From the calculated results seen in figure 3, step–dislocation interactions are quite plausible as well as dislocation–surface domain interactions.

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