Si(100) Surface under an Externally Applied Stress

F. K. Men, W. E. Packard,[a] and M. B. Webb
Physics Department, University of Wisconsin, Madison, Wisconsin 53706
(Received 11 July 1988)

Loading a cantilevered bar allows studying the effect of a uniform, externally applied, and continuously variable strain field on the properties of surfaces. Straining a nominally flat Si(100) surface produces unequal populations of the 2×1 and 1×2 domains. The domain compressed along the dimer bond is favored. The effect depends on the strain and not the strain gradient. The effect saturates at a strain of 0.1% when 90% of the surface is in the favorable domain. The kinetics of developing and annealing away the asymmetry are identical and thermally activated.

PACS numbers: 68.35.Bs, 68.35.Gy

It is clear from both theory and experiments that strain is an important ingredient in the physics underlying the reconstruction of surfaces. A number of authors[1] have used Keating-type analyses where stretching and bending force constants are assigned to bonds and a structure of a given topology is optimized by minimizing the elastic energy. Needs[2] and Vanderbilt[3] have calculated surface stress tensors from geometry-optimized total-energy calculations. In beautiful experiments, the importance of strain has been demonstrated in Si-Ge alloys of different compositions and strained epitaxial overlayers of different thicknesses where different reconstructions are observed for different average strain.[4]

It would be useful to be able to apply externally a uniform and continuously variable strain, i.e., to have strain as an independent variable. With such a capability one would hope to do a number of interesting experiments including the following: determination of the strain-temperature phase diagram for reconstructed surfaces, lift the surface energy degeneracy of orientational domains and perhaps prepare single-domain surfaces, study the effect of strain on surface segregation in alloys with atoms of different sizes, alter the natural misfit for incommensurate overlayers, and study the effect of strain on surface magnetism.

We have mocked up several mechanical schemes. Here we report experiments using the simplest of these, which is to load the free end of a cantilevered bar. This produces a uniaxial strain at the surface varying linearly along the length of the bar. In this paper we take the "strain" to be that calculated at the surface from the elastic theory for the cantilevered bar; we have not yet measured the surface lattice parameter with sufficient precision to determine the strain directly.

Our first experiments have been on the Si(100) 2×1 surface which reconstructs by dimerization of atoms in adjacent rows. In areas of the surface separated by an odd number of monatomic steps the orientation of the dimer bonds is rotated by 90° giving 2×1 and 1×2 orientational domains which give distinct $\frac{1}{2}$-order superlattice LEED reflections.[5]

The samples are 0.3 mm thick, 2 mm wide, and 19 mm long. Twenty samples are clamped in a carousel mounted on a rotary feedthrough. The carousel is rotated to bring the free end of a sample into the gap between two carefully machined and aligned wedge-shaped Ta anvils. The anvil assembly moves up or down on a high-precision micrometer to bend the sample. A 0.8-mm deflection of the anvil corresponds to 0.1% strain at the fixed end of the bar. Different samples break at different strain, but we frequently achieve 0.3% strain at room temperature. We check for plastic deformation by measuring the position of the end of the bar after removing the load.

The first experiments were on nominally flat surfaces which originally have equal populations of the two domains as indicated by the equal intensities of the $(0,\frac{1}{2})$ and $(\frac{1}{2},0)$ reflections at normal incidence. Straining the crystal at room temperature causes no change because of the slow kinetics. However, straining the sample at higher temperatures produces unequal intensities. In Fig. 1 we show the intensities of two super-

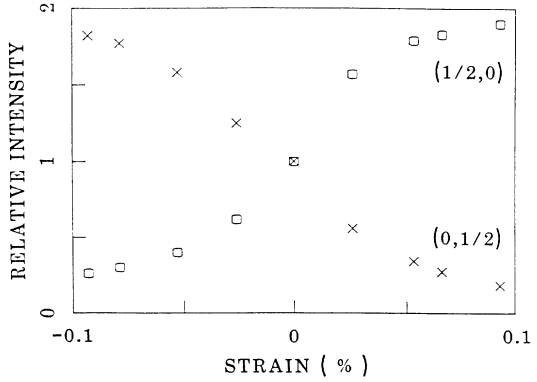

FIG. 1. The ratio of the intensity of the $(\frac{1}{2},0)$ and $(0,\frac{1}{2})$ LEED beams to their values at zero strain plotted as functions of the calculated surface strain. The data were measured at a fixed position along the bar for various deflections of the end. The domain compressed along the dimer bond is favored.

lattice beams for different deflections of the end of the bar plotted as functions of the calculated surface strain at the position of the incident beam. For these data the intensities were measured after the sample was strained at an elevated temperature and then cooled under load. The same relative intensities are measured at the elevated temperature—both decreased by the Debye-Waller factor. If the load is relieved at room temperature the same asymmetry remains.

The intensities indicate unequal populations of the two domains. The sense is that the domain compressed along the direction of the dimer bond is favored. The asymmetry saturates at about 0.1% strain when approximately 90% of the surface is in the favorable domain (see below). Angular profiles of the superlattice reflections remain instrument limited at all incident electron energies, indicating that even the minority domains have average terrace widths of greater than 500 Å.

Figure 2(a) shows similar data but measured by moving along the length of the sample at each of a number of fixed deflections of the end. These data indicate that the asymmetry depends on the strain and not the strain gradient along the bar.

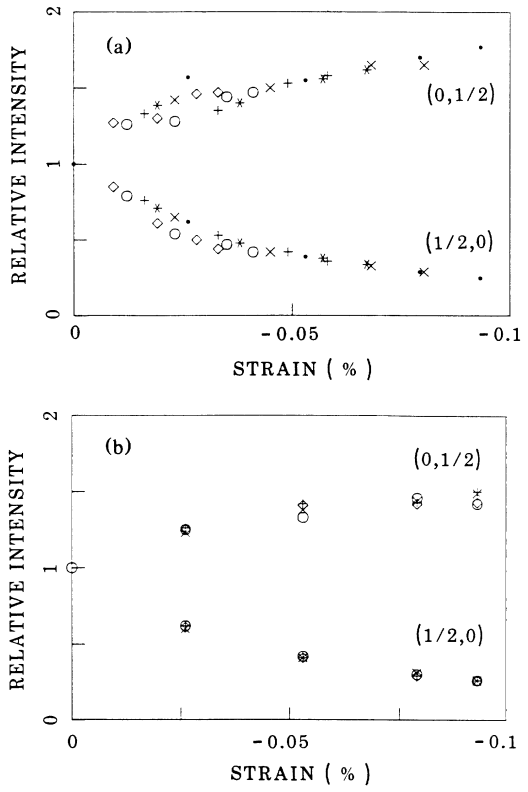

FIG. 2. (a) The relative intensities of the superlattice reflections as functions of calculated surface strain measured by moving along the length of the bar for given deflections of the end. Each symbol is the data for a given deflection. The maximum strain for a particular deflection is given by the termination of the data points with that symbol. (b) The steady-state relative intensities of the superlattice reflections measured for the sample strained at various temperatures indicated by the different symbols. The temperatures ranged from 550 to 800 °C.

The kinetics to develop or remove the asymmetry depend on the temperature, but the steady-state asymmetry depends only on the strain and not on the temperature. This is shown in Fig. 2(b) where the steady-state intensities are shown as functions of strain from experiments where the bar was loaded at a number of different temperatures. This suggests that the effect is due to the mechanical energy rather than the free energy.

The kinetics are illustrated in Fig. 3 which shows the intensity of one of the reflections as a function of time after applying and removing the load all at a fixed temperature. The kinetics are well fitted by $\Delta I(t) = \Delta I(\infty)(1 - e^{-t/\tau})$ and the time constant is the same in both directions. Time constants vary from 24 sec at 670 °C to 115 sec at 550 °C. As a function of temperature the time constant follows an Arrhenius plot with an activation energy of about 0.85 ± 0.15 eV (the large uncertainty is due to the uncertainty in the temperature measurements in these first experiments). Changing the domain populations requires motion of steps and surface mass transport, and so this activation energy may be reasonable.[6]

The experiments have been repeated on a number of different samples cut from each of two wafers with essentially the same results. The fraction of the surface in the majority domain at saturation has varied from 84% to 91% between samples but is constant within 2% for repetitions of the experiment on a given sample. There are differences between tension and compression of a few percent, but we have observed these small differences in both directions. The effect is very sensitive to surface contamination and is inhibited by exposure to the residual gas. In view of this it is not certain that the saturation is an intrinsic property.

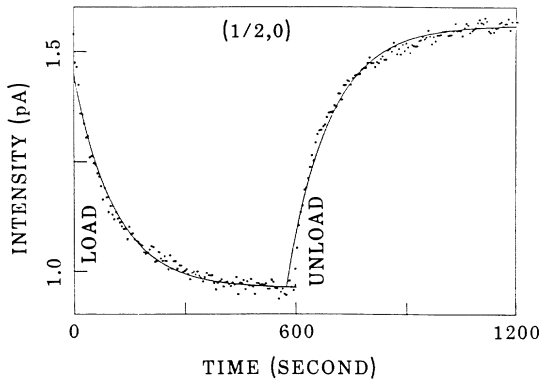

FIG. 3. The intensity of the $(\frac{1}{2},0)$ superlattice reflection as a function of time after applying and removing the external compressive stress. These data were taken at 550 °C. The time constant is 114 ± 7 sec.

In the above experiments, all strains were within the elastic limit as shown by the position of the sample end after removing the load. In one experiment a sample was straining at 900 °C for 30 min which produced plastic deformation. (The sample was loaded to a deflection of the end of 0.7 mm and a 0.5-mm deflection remained after unloading.) The asymmetry in the domain populations of this deformed sample could not be completely removed by annealing the unloaded sample. Repeating the strain experiment with this deformed sample gives similar results as shown in Fig. 1 except that equal populations now occur with an applied stress opposite to that which caused the plastic deformation. (In this case the domain populations were equal at an applied strain of −0.016%.) In a sense this is counterintuitive; the plastic deformation tends to relieve the applied stress and after unloading one might think that there would be a residual strain of opposite sign.

Clearly the step configuration must change as the asymmetry is developed or annealed and it would be useful to characterize the steps. In the above experiments on flat samples, the terraces are sufficiently large that we cannot detect them in our experiments. Therefore we have attempted experiments with purposely large step densities by looking both at sputtered and then partially annealed surfaces and at vicinal surfaces miscut by 4°. In the former, the heating necessary to develop the asymmetry also anneals away the additional steps and the surface behaves just as the nominally flat surfaces did. In the latter, after cleaning and annealing, the unloaded vicinal surface is about 90% in a single domain with primarily double steps. (The minority superlattice reflections are very broad, indicating minority terraces very narrow in the direction perpendicular to the nominal step edges.) We have strained this surface along, perpendicular to, and at 45° to the step edges. In all cases there is no change in the superlattice reflections with strain.

We presently have no understanding of the mechanism of this effect. If it were just that the applied stress lifts the surface energy degeneracy of the two domains, it is difficult to understand the identical kinetics for the development and annealing of the unequal populations. After removing the applied stress the only driving force for annealing would be entropic. It is also difficult to understand the saturation if it is indeed intrinsic. It seems unlikely that the saturation is due to direct interaction between the steps since the minority-domain terraces remain several hundred angstroms wide. One might consider the interaction between the applied strain and the strain field associated with the steps. It is observed that the effect depends on the applied strain and not the gradient. Whereas a uniform strain will not cause an individual step to translate, it does exert a torque and can alter the configuration of steps. Phenomenologically the behavior is like a two-layered system, the selvage and the substrate with different natural lattice parameters. The steps then somehow adjust to a configuration which minimizes the total energy and this configuration leads to equal domain populations under zero external stress but unequal populations under load.

This work was supported by NSF Grant No. DMR-8720778. We thank D. Vanderbilt for stimulating discussions, L. C. Feldman for the vicinal sample, and the Silicon Sensors Corp. for cutting wafers.

[a]Present address: Physics Department, University of Notre Dame, Notre Dame, IN 46556.

[1]P. N. Keating, Phys. Rev. **145**, 637 (1966); J. A. Appelbaum and D. R. Hamann, Surf. Sci. **74**, 21 (1978); R. M. Tromp, Surf. Sci. **155**, 432 (1985); G. X. Qian and D. J. Chadi, Phys. Rev. B **35**, 1288 (1987).

[2]R. J. Needs, Phys. Rev. Lett. **58**, 53 (1986).

[3]D. Vanderbilt, Phys. Rev. Lett. **59**, 1456 (1987).

[4]H. J. Grossmann *et al.*, Phys. Rev. Lett. **55**, 1106 (1985); A. Ourmazd *et al.*, Phys. Rev. Lett. **57**, 1332 (1986), and references therein.

[5]For a review of the structure and properties of the Si(100) surface see D. Haneman, Rep. Prog. Phys. **50**, 1045 (1987). For a discussion of models of the steps on Si(100) see D. J. Chadi, Phys. Rev. Lett. **59**, 1691 (1987), and for observations of steps on Si(100) see T. Nakayama, Y. Tanishiro, and K. Takayanagi, Jpn. J. Appl. Phys. **26**, L280 (1987); P. E. Wierenga, J. A. Kubby, and J. E. Griffith, Phys. Rev. Lett. **59**, 2169 (1987), and references therein.

[6]We know of no definitive measurements of the surface diffusion on Si(100) but for observation of the ordering kinetics on Si(111) with an activation energy of 1.1 eV see S. M. Bedair, Surf. Sci. **42**, 595 (1974).