PHYSICAL REVIEW B VOLUME 24, NUMBER 6 15 SEPTEMBER 1981

Nature of vicinal laser-annealed Si(111) surfaces

Y. J. Chabal, J. E. Rowe, and S. B. Christman
Bell Laboratories, Murray Hill, New Jersey 07974
(Received 18 March 1981)

Low-energy electron diffraction studies of vicinal Si(111) surfaces cut along the [11$\bar{2}$] direction show that a regular array of steps is formed upon laser annealing, with step height (3.06 Å) somewhat less than the double-layer separation (3.14 Å) and edge atoms threefold coordinated. For energy densities between 0.2 and 0.5 J/cm^2 at 5320 Å a weak buckling reconstruction is present and can be aligned by the step edges. Thermal annealing to 300° C allows first the atoms in the neighborhood of the step edges to relax, resulting in a small increase of the step height. Further annealing above 400° C results in the growth of the terrace widths and simultaneous increase of the step height. The 7×7 reconstruction is observed as soon as the terrace width is large enough to support several surface unit cells (> 200 Å). Our results indicate that the high-temperature equilibrium configurations for vicinal Si(111) surfaces consist of a regular array of monatomic steps ($d \sim 3.1$ Å) and can be quenched by laser annealing into a metastable phase. The low-temperature phase displays an effective attractive step-step interaction resulting in the formation of very large terraces and large risers. The nature of this attractive interaction is discussed.

I. INTRODUCTION

The influence of steps on the electronic properties,[1,2] reactivity,[3] and chemical activity[4] of the Si(111) surface has been well recognized. In a more fundamental way, steps could be a key feature in the 7×7 reconstruction[5] as was recently shown by He diffraction results[6] and low-energy electron diffraction (LEED) analysis[7] of clean and H-covered Si(111) 7×7 surfaces, respectively. Unfortunately, monatomic steps are not present on well-annealed vicinal Si(111) surfaces at room temperature. Instead, large terraces are formed with the characteristic 7×7 reconstruction present. Only on more poorly characterized cleaved[1] or ion-etched[4] surfaces can such monatomic steps be observed. We have therefore prepared vicinal Si(111) surfaces by laser annealing,[8] resulting in the reproducible formation of a regular array of steps. The LEED intensity angular profiles and their voltage dependence are well explained by considering only single kinematic scattering off the top layer,[9] which gives precise information on the spatial distribution of the step atoms. This preparation technique also allowed us to verify that the short-range buckling reconstruction previously found on flat laser-annealed[10] Si(111) is oriented by the steps, as is the case for cleaved Si(111) 2×1 surfaces. The room-temperature structure of the laser-annealed phase, however, is a metastable quenched-in phase of the high-temperature equilibrium configuration. A long thermal annealing to 800 °C followed by slow cooling restores the equilibrium configuration at room temperature consisting of large terraces with 7×7 reconstruction and tall risers. By following the LEED angular profiles at intermediate annealing temperatures (< 600 °C) we find that a small initial relaxation of the steps is followed by a growth of terraces and discrete increases in step height. The observed equilibrium attractive interaction between steps at low temperatures is discussed in terms of various thermodynamic models.

II. EXPERIMENTAL

The Si(111) samples (n type, $\rho = 10\ \Omega$ cm) were cut at an angle α from the (111) plane ($\alpha = 2°$, 3°, and 5°) and cleaned by argon sputtering followed by thermal annealing to 1000 °C. The LEED pattern obtained after such a treatment was a sharp 7×7 pattern with no indication of spot splitting. Auger analysis showed that less than 1% of an impurity monolayer was present. The laser annealing was performed by irradiating the sample with a series of pulses (0.4 J/cm^2 in 10-nsec pulses at 530 nm) originating from a Nd:YAG laser equipped with a frequency doubler. LEED angular profiles were recorded from a standard commercial LEED

screen by a vidicon camera interfaced to a minicomputer.[11] The angular resolution of the vidicon camera can be defined as the probed area of width equivalent to $\Delta\theta_x = 0.1°$ and height $\Delta\theta_y = 2°$. The sample normal was tilted with respect to the electron gun axis by about 11° so that the (00) beam could be investigated as well as the nonspecular beams. The UHV chamber[12] was also equipped with a discharge lamp and an electron analyzer so that photoemission spectra could be obtained.

III. ANALYSIS OF THE LASER-ANNEALED SURFACES

Upon laser annealing with energy densities larger than 0.2 J/cm^2, the samples displayed a 1×1 LEED pattern with sharp spot splittings at specific voltages as summarized in Table I (middle column). The energies at which a given spot (hk) is single or split give specific information on the step height and the nature of the steps.[1,9]

Based on a kinematical treatment of single scattering from the top layer, the energy at which a spot (hk) is split is[13]

$$E_n(hk) = \left|\frac{\hbar^2}{8md^2}\right| \left\{\frac{(n - xh - yk)^2 + (h\vec{a}^* + k\vec{b}^*)^2 d^2}{(n - xh - yk)\cos\theta - (h\,|\vec{a}^*|\cos\phi_a + k\,|\vec{b}^*|\cos\phi_b)d\sin\theta}\right\}^2, \tag{1}$$

for $n = (m + \frac{1}{2})$ where $m = 1,2,\ldots$. For $n = m$, the (hk) spot is not split since the contribution from the array of terraces adds in phase to that of the individual atoms of each terrace and thus a strong single spot is observed at an angular position equal to the average of the split spots. This treatment is also correct when multiple-scattering dynamic corrections are included as long as only intrastep scatter-

TABLE I. Laser-annealed Si(111) 5°. Theoretically and experimentally determined electron energies (in eV) for which the LEED spots are split or single. The dashes represent energies where the spots have too weak an intensity or are not visible to the camera to determine the spot shapes. $\theta = 11°$, $\gamma_a = 60°$, $\gamma_b = 0°$, $d = 3.06$ Å, and $|\vec{a}| = 3.84$ Å.

		Theory $[11\bar{2}]$		Experimental voltages		Theory $[\bar{1}\bar{1}2]$	
(hk)	n	Single	Split	Single	Split	Single	Split
(00)	1.5		9		9		9
	2	17		14		17	
	2.5		26		24		26
	3	37		35		37	
	3.5		51		51		51
	4	67		66		67	
	4.5		84		86		84
$(0\bar{1})$	1.5		18		-		23
	2	26		26		32	
	2.5		36		38		44
	3	48		50		57	
	3.5		62		-		73
$(\bar{1}0)$	1.5		25		-		20
	2	35		35		28	
	2.5		46		46		38
	3	60		59		50	
	3.5		76		-		65

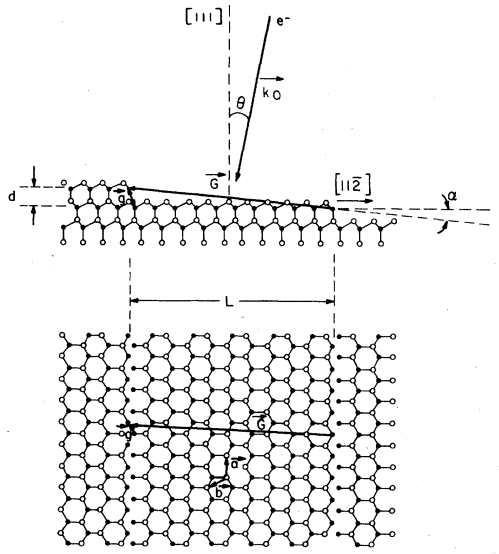

FIG. 1. (a) Side view of the terrace (along the $[1\bar{1}0]$ direction), characterized by the translation vector $\vec{G} = (N_a + x)\,\vec{a} + (N_b + y)\,\vec{b} + d\hat{c}$. The step direction is $[11\bar{2}]$ and the optical surface (parallel to $\vec{G}$) makes an angle α with the terrace plane (111). The electrons are incident in a plane defined by [111] and $[11\bar{2}]$ with a polar angle θ. For the surface shown, $\alpha = 5°$. (b) Top view of the terrace (along the $[\bar{1}\bar{1}1]$ direction) where the translation vectors $\vec{a}$ and $\vec{b}$ are defined. For this terrace, $N_a = 5$ and $N_b = 10$. The translation vector $\vec{g}$ is $(\frac{2}{3},\frac{1}{3},d)$ in the coordinate system $(\vec{a},\vec{b},\hat{c})$ where $\hat{c}$ is a unit vector along the [111] direction. The terrace length L is 35 Å ($\alpha = 5°$).

ing is dominant. Since the step widths are large, ~ 35 Å compared to electron scattering lengths $\sim 5-8$ Å, we expect interstep multiple scattering to be negligible compared to intrastep multiple scattering. The surface reciprocal vectors are defined by

$$\vec{a}^* = \frac{\vec{b} \times \hat{c}}{\vec{a} \cdot (\vec{b} \times \hat{c})} \tag{2}$$

and

$$\vec{b}^* = \frac{\hat{c} \times \vec{a}}{\vec{a} \cdot (\vec{b} \times \hat{c})} ,$$

where, for the Si(111) surface,

$$|\vec{a}^*| = |\vec{b}^*| = (2/\sqrt{3})(3.84)^{-1}\,\text{Å}^{-1} . \tag{3}$$

The parameters ϕ_a and ϕ_b are the azimuthal angles of the incident electron wave vector with respect to the reciprocal vectors $\vec{a}^*$ and $\vec{b}^*$, respectively. The local structure of the step is contained in the vector $\vec{g} = x\vec{a} + y\vec{b} + d\hat{c}$ joining an edge atom to the closest equivalent atom in the adjacent terrace as shown in Fig. 1. While the modulation of the (00) beam is only sensitive to the step height d, the voltage dependence of nonspecular beams will in addition depend on the translational nature of the step. For Si(111), steps along the $[11\bar{2}]$ direction for which the edge atoms have three nearest neighbors (one dangling bond) are characterized by $x = \frac{2}{3}$ and $y = \frac{1}{3}$, while steps along the $[\bar{1}\bar{1}2]$ direction for which the edge atoms have only two nearest neighbors (two dangling bonds) are characterized by $x = \frac{1}{3}$ and $y = \frac{2}{3}$. Table I gives the theoretical predictions of Eq. (1) for both types of steps and summarizes the experimental observations for a 5° sample. Our LEED data shows clearly that our laser-annealed vicinal surface is made up of a regular array of terraces with monatomic steps oriented in the $[11\bar{2}]$ direction, expected from x-ray orientation prior to cutting the sample. We note that optical reflection measurements show that the average inclination of the surface remains the same no matter what heat treatment is performed, either laser quenching or thermal annealing.

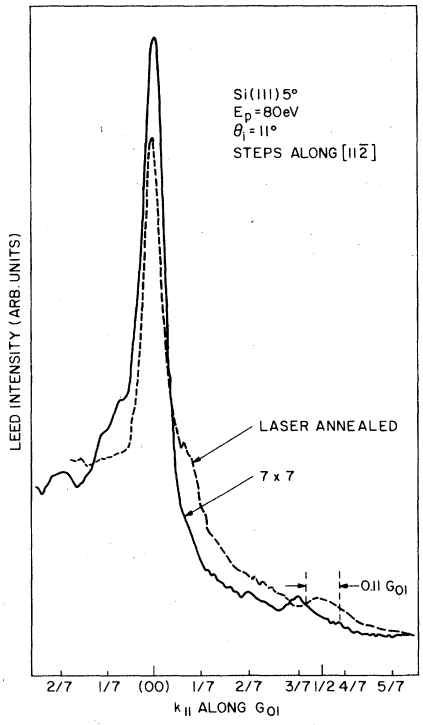

FIG. 2. Angular profile of the (00) spot along the ⟨01⟩ direction for $E_p = 80$ eV. The width of the half-order streak is 0.11 G_{01}.

We first note that the best fit to the voltage dependence of the (00) beam gives a step height 2.5% smaller than the bulk double layer distances ($d_0 = 3.14$ Å). This apparent contraction was reported for cleaved and sputtered Si(111) surfaces with monatomic steps along the $[\bar{1}\bar{1}2]$ direction,[1,4] and attributed to a tilt in the average terrace plane with respect to the (111) plane. We will show that this tilt is probably due to stresses caused by cleaving, sputtering, or laser annealing. The magnitude of the splitting (e.g., $\frac{1}{9}G_{01}$, for the 5° sample) is consistent with a terrace width $L = d/\tan\alpha \approx 35$ Å. Next, we have investigated the weak LEED background around the half-order position. For flat laser-annealed Si(111) prepared in similar conditions, the width of the half-order spots $\sim 0.25G_{01}$ indicated that the domains of 2×1 symmetry were smaller than 16 Å.[10] If the buckling reconstruction is the same in nature as that of the cleaved Si(111), then these domains should be aligned by a regular array of steps. In Fig. 2, the half-order streak observed at $G = (1\frac{1}{2})$ on vicinal laser-annealed Si(111) is shown. Its width is $\sim 0.11G_{01}$, consistent with domains as large as the terrace width ($L = 35$ Å), i.e., over twice as large as the domains formed on the flat Si(111) surfaces.[10] We note that the half-order intensity only appears along the directions perpendicular to the step edges indicative of a good domain alignment by the step array.

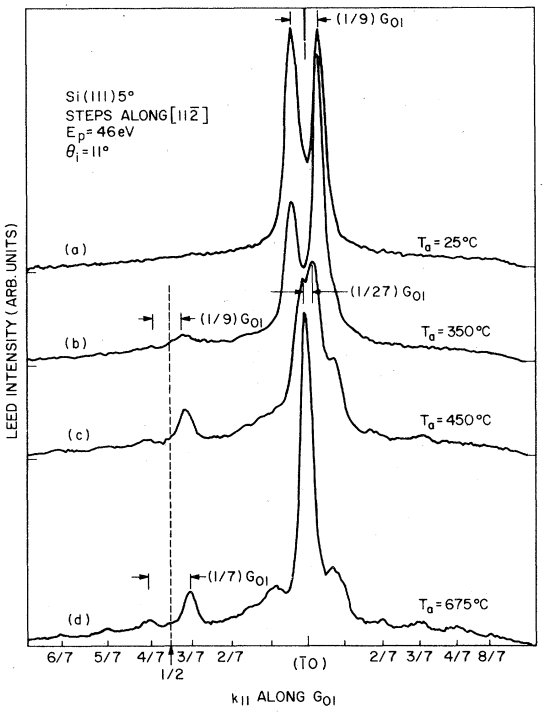

FIG. 3. Angular profile of the $(\bar{1}0)$ spot along the $\langle 01 \rangle$ direction for $E_p = 46$ eV. The temperature T_a corresponds to the thermal-annealing temperature to which the sample was subjected for 60 sec subsequent to laser anneal.

IV. STABILITY OF LASER-ANNEALED VICINAL SURFACES

The stability of the regular array of steps was investigated by subjecting the laser-annealed sample to a series of thermal-annealing cycles to higher and higher temperatures. Some of the resulting angular profiles of the $(\bar{1}0)$ spot are shown in Fig. 3. In curves (a) and (b), the splitting of the integral order spot ($G = \bar{1}0$) is $\sim \frac{1}{9}G_{01}$ indicating a terrace width of ~ 35 Å. However, the asymmetry in curve (b) is a result of step relaxation and can be explained by the simple kinematical theory either by letting d be the double layer distance, $d_0 = 3.14$ Å, instead of the contracted distance, $d = 3.06$ Å, or by decreasing the x component of the vector $\vec{g}$ by $\sim 20\%$. The latter would indicate a general motion of the step atoms parallel to the step which seems unlikely. A more plausible explanation would involve a slight relaxation of the terrace, i.e., an elimination of the 2.5% contraction, so that the average terrace plane is now in perfect registry with the (111) plane. The origin of the original contraction may therefore be associated with strains (introduced by the fast quenching of the surface) which are suppressed by a very mild annealing (350 °C for 1 min). The strain fields partially inhibit the formation of a 2×1 reconstruction as shown by the absence of the half-order spot in curve (a) for $E_p = 46$ eV. For the strain-free surface of curve (b), the half-order spot appears but is split asymmetrically with the same splitting as that of the integral order spot, as expected for kinematical theory. The absence of half-order spot in curve (a) is very similar to the behavior of cleaved 2×1 Si(111) surfaces where no reconstruction can be seen by LEED, at low voltages, when the terrace width is less than 30 Å,[14,15] i.e., when the strain fields affect most of the terrace area. We note that when the half-order streaks cannot be seen at $E_p = 46$ eV it can still be seen at $E_p = 80$ eV as was shown in Fig. 2. Some 2×1 is therefore still present but with enough strain that the half-order spots undergo a destructive interference at low energies. The strain fields do not suppress completely the buckling reconstruction but

affect the spacings enough that very little contribution goes into the half-order streaks at low electron energies (< 55 eV).[10]

Curve (c) is obtained after an intermediate annealing and shows a splitting with a magnitude $\sim \frac{1}{3}$ of the original splitting present in curves (a) and (b) characteristic of terraces ~ 100 Å wide. Again, Eq. (1) predicts such a splitting for Si(111) cut in the $[11\bar{2}]$ direction if $d = 3d_0$ and correspondingly $x = 0$ and $y = 1$. The natural structure of the riser is the $(11\bar{1})$ plane making an angle of 109.5° with the (111) terraces. Such a riser would be very stable. In contrast, a step oriented towards the $[\bar{1}\bar{1}2]$ direction would form a riser containing a (001) plane which makes an angle of 125.5° with the (111) terraces. The stability of these two kinds of steps is therefore not necessarily identical but there is yet no detailed experimental comparison. The overall behavior for laser-annealed vicinal surfaces is the same for both types of steps.[8] In both cases the single-step array formed upon laser annealing disappears upon thermal annealing to 800 °C to allow the growth of very large terraces where the 7×7 reconstruction is present. We note that for our experimental parameters ($E_p = 46$ eV, $\theta = 11°$), the contribution to the $(\bar{1}0)$ spot from double-height steps ($d = 6.1$ Å, $L = 70$ Å) is expected to give a single diffraction spot (no splitting). Such a spot is not clearly seen in our data obtained between curves (b) and (c) in Fig. 3, but we cannot claim that such a double-height step as an intermediate configuration does not occur. In fact, such double steps were observed on sputtered Si(111).[4] Our data rather indicate that a mixture of single, double, and triple steps can be formed for intermediate annealing temperatures. As the sample is further annealed to 675 °C, the $(\bar{1}0)$ spot sharpens up with a width consistent with domains wider than 200 Å, and the 7×7 reconstruction, first apparent in curve (c) is well developed in curve (d).

Schematically, the whole process can be summarized in Fig. 4 where the surface free energy is plotted versus some generalized configuration coordinate Q which depends on the step density, $(\tan\alpha)/h$. For a given vicinal surface, the inclination α is fixed and the highest step density corresponds to a regular array of monatomic steps of height $h = d_0$. In the high-temperature limit [curve (a)] which can be reached by laser heating, the effective step interaction is repulsive and the free energy, dominated by the entropy, is lowest at the highest step density (point B in Fig. 4). In the low-temperature limit [curve (b)], the free-energy curve is higher since less

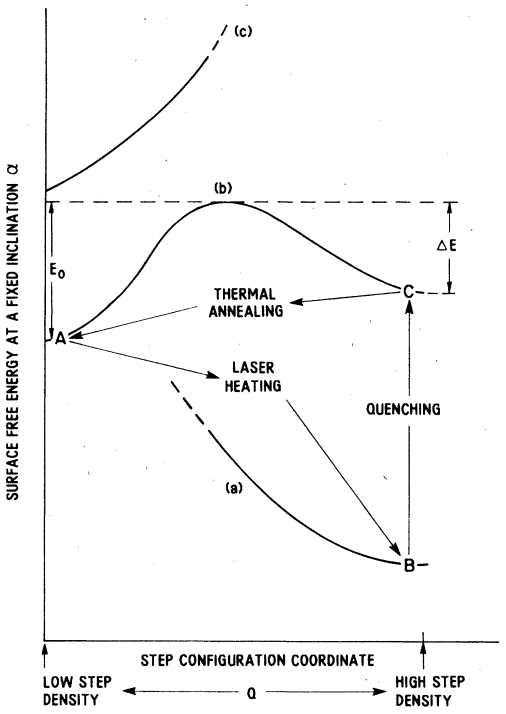

FIG. 4. Schematic representation of the free energy associated with the vicinal surface as a function of step density for two different temperature regimes. Curve (b) is the free-energy curve associated with the low-temperature regime, while curve (a) is associated with the high-temperature regime. Curve (c) represents the effect of surface reconstruction on the free energy.

entropy is available. The high step density position (point C) is a local minimum, i.e., a metastable position which can be reached by very rapid quenching. The absolute minimum is at the very low step density end (point A), corresponding to large terraces. Within this equilibrium picture, the activation energy can be estimated from the formula $\Delta t = \tau_0 e^{\Delta E/kT}$, where Δt is the annealing time and τ_0 the inverse of the attempt frequency which we take to be $\tau_0 \sim 10^{-13}$ sec. For our 60-sec annealing times, the temperature at which the 7th-order spots appear is $T \sim 675$ K. The activation energy is $\Delta E \sim 2$ eV. After the thermal annealing, no splitting in the LEED spot was observed for temperatures below 800 °C in contrast to the findings of Olshanetsky and Shklyaev for an inclination $\alpha = 8°$.[4] Since the high step density cannot be recovered without annealing above 1130 K, we estimate that $E_0 \gtrsim 1.6\ \Delta E$.

The nature of the attractive interaction at low temperatures could be dominated by the covalent

reconstruction of the terraces or even of the large risers or by elastic forces. Although this question cannot be answered at present, the equilibrium configuration of surfaces has been the subject of many theoretical considerations[16,17] which could shed light on this problem. For Cu(001), the general approach of Cabrera[16] was followed by Wynblatt[18] who chose a pairwise interaction between atoms according to a Morse potential. By considering a surface with two ledges and allowing the outermost layer to relax, he found that sequential steps are always repulsive, for all T. Moreover, the effective step interaction due to the interaction of the displacement fields was found to be short range. This treatment gives reasonable results for vicinal Cu(001) but clearly fails for Si(111) or other semiconductor surfaces. Blakely and Schwoebel[19] used a phenomenological approach to relate the interaction between pairs of steps to the surface tension and find that such an interaction is long range and always repulsive for like steps. This model may be appropriate for the high-temperature region of Si(111) but is incomplete for the low-temperature region. Gruber and Mullins[20] pointed out the importance of finite temperature calculations where entropy effects become important. At high temperatures, the wandering of the ledges causes an additional "hard-core" step-step repulsion since adjacent steps cannot cross one another.

However, if there is also an attractive "pinning" potential favoring multiple height or bunched steps there is a competition between this entropy-driven repulsion favoring separated steps and the pinning energy. Chui and Weeks[21] have suggested that the bunched step system offers a physical realization of the pinned roughening transition found by Abraham[22] in the two-dimensional Ising model. This approach shows that there should be a phase transition with the risers breaking apart at some well-defined temperature T_R.[23] A physical explanation for this pinning energy could result from surface reconstruction as suggested by Rode's observation[24] that multiple height steps are formed mostly on surfaces which reconstruct.[25] Schematically, the part of the free energy associated with reconstruction can be represented by curve (c) in Fig. 4. For low step density, a large percentage of terrace area is reconstructed and the free energy is lowered. Indeed, there is evidence[14,15] that surface reconstruction does not occur in the immediate vicinity of steps.

So far, kinetic effects have been neglected. It is expected, though, that mass transport properties will, in general, be an important factor in the structure of vicinal surfaces.[26–30] For silicon, an estimate for the diffusion rate on the (111) surface can be obtained from the work of Csepregi *et al.*,[31] who found that the epitaxial regrowth of amorphous Si on an underlying silicon substrate is ~ 1 Å/min at 450 °C along the [100] direction with an activation energy of 2.35 eV. Since for Si(111) surface diffusion rates about 10^5 times faster than bulk growth have been observed at 1200 °C,[32] we estimate the activation energy for surface diffusion to be about 1 eV. Correspondingly, surface diffusion rates of 0.3 Å/min at room temperature, 0.5 μ/min at 100 °C, and 1 cm/min at 500 °C are expected. Surface atoms are therefore quite mobile on Si(111) so that equilibrium configuration can be reached within the laboratory times (few minutes) above 200 °C, which we still consider as the low-temperature regime. Therefore the equilibrium approach discussed earlier should account for most of the features.

Finally, the influence of impurities on the vicinal surface structure should be considered.[33] Indeed, Isa *et al.*[34] found that 0.2 monolayer of Ca could stabilize monatomic steps on Au. It is possible, therefore, that the double step structure observed by Olshanetsky and Shklyaev[4] below 800 °C is due to a very small amount of impurities such as in the case of UO_2.[9] The process of laser annealing, however, has been shown to clean the surfaces,[35] as was confirmed for our samples by Auger analysis ($< 5 \times 10^{-3}$ monolayer of impurities). The results obtained on laser-annealed vicinal surfaces are therefore probably intrinsic to the reconstructed Si(111) surface.

V. CONCLUSION

By preparing vicinal Si(111) surface by laser annealing, we have been able to quench the high-temperature structure dominated by an entropy induced repulsive interaction into a metastable phase at room temperature. In agreement with Olshanetsky and Shklyaev[4] we have shown that steps with a double layer height can be formed along the $[11\bar{2}]$ direction. From the work of Zehner *et al.*[8] it is clear that this situation is similar for steps pointing in the $[\bar{1}\bar{1}2]$ direction, although the local nature of the two types of steps is quite different, resulting in a preference for the $[\bar{1}\bar{1}2]$-type steps on cleaved Si(111).[1,36] The two metastable laser-annealed and cleaved vicinal surfaces have been shown to behave in a similar fashion. Although laser-annealed surfaces are less ordered than cleaved surfaces, the

same type of reconstruction takes place and can be aligned by steps. The temperature range at which the terraces grow and the 2×1 reconstruction disappears is similar in both cases. Strain fields associated with steps tend to lower the effective step height and to prevent reconstruction in the immediate vicinity of the step for both surfaces.

With regard to the stability of vicinal Si(111) surfaces, it seems clear that a proper model should account for an attractive step interaction at low temperature: The high-temperature regime, probably dominated by entropy, is characterized by an effective repulsive interaction. Further studies should help determine if the temperature at which the interaction becomes repulsive is lower than the order-disorder transition temperature for the 7×7 reconstruction.[37]

ACKNOWLEDGMENTS

We would like to acknowledge many useful and stimulating discussions with T. M. Rice who emphasized the importance of considering step interactions and the entropy contribution to the surface free energy. Additional discussions were also contributed by J. D. Weeks, E. G. McRae, and M. J. Cardillo. We would like to thank C. V. Shank for the loan of the doubled Nd:YAG laser and D. A. Zwemer for help with many of the measurements.

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