HRLEED AND STM STUDY OF MISORIENTED Si(100) WITH AND WITHOUT A Te OVERLAYER

ANL/MSD/CP--90538
CONF-961202--55

Salima Yala and Pedro A. Montano

Materials Science Division, Argonne National Laboratory, Argonne, IL 60439
and
Department of Physics, University of Illinois-Chicago, Chicago, IL

Materials Science Division
Argonne National Laboratory
Argonne, IL 60439

December, 1996

/jc

Distribution:

1-2. M. J. Masek
3. B. D. Dunlap
4. P. A. Montano
5. F. Y. Fradin
6. R. Gottschall
7. Editorial Office
8. Authors

Submitted to symposium proceedings of the Materials Research Society Fall Meeting, December 1996.

This work is supported by the Division of Materials Sciences, Office of Basic Energy Sciences of DOE, under contract No. W-31-109-ENG-38.

HRLEED AND STM STUDY OF MISORIENTED Si (100) WITH AND WITHOUT A Te OVERLAYER

SALIMA YALA AND PEDRO A. MONTANO
Materials Science Division, Argonne National Laboratory, Argonne IL 60439,
pedro_montano@qmgate.anl.gov
Department of Physics, University of Illinois-Chicago, Chicago, IL

ABSTRACT

The growth of high quality Te on misoriented Si(100) is important as an intermediate phase for epitaxial growth of CdTe. The misorientation angle plays a key role in the growth quality of CdTe/Si(100); this incited our curiosity to investigate the effect of the misorientation angle on the topography of the surface structure of Si(100). Our main goal is to show the relation between the misorientation angle, the terrace width and the step height distributions. HRLEED (High Resolution Low Energy Electron Diffraction) provides information in reciprocal space while STM gives real space topographic images of the surface structure. STM and HRLEED measurements were performed on Si(100) with misorientation angle $\vartheta = 0.5°$, 1.5° and 8° towards the [110] direction and $\vartheta = 4^0$ towards the [130] direction. Except for the 8° misorientation in which case a regular step array with diatomic step height was observed, for the other misorientations the terrace width was variable. The average terrace width decreased with increasing misorientation angle. A mixture of diatomic and monatomic step heights was observed on the 0.5° and 1.5° misoriented Si(100) samples. It proves that one can not assume purely monatomic step height for low misorientation angles. Our results do not agree with the belief that at low miscut angle A and B terraces are equal and that as the misorientation angle increases the B terrace tends to be wider than the A terrace. In fact, pairing of terraces was not observed at all. Te was deposited at a substrate temperature of 200 C. We observed a significant reduction in the terrace widths for all miscut angles.

INTRODUCTION

In recent years, the growth of high quality thin films became first priority because of the need to miniaturize microelectronics. Among all the substrates, Silicon is the most attractive one not only for its high crystalline properties but also for its availability and low cost in the market. The growth of high quality CdTe on misoriented Si(100) represents a challenging and yet promising project for technological applications (substrate fror growth of MCT, for x-ray detectors) [1-3]. The misorientation angle plays a key role in the growth quality of CdTe/Si(100); this incited our curiosity to investigate the effect of the misorientation angle on the topography of the surface structure of Si(100). Our main goal is to show the relation between the misorientation angle, the terrace width and the step height distributions. Si(100) with $\vartheta = 0.5°, 1.5°, 4°$ and 8° misorientation angles towards [110] and [130] were systematically investigated using HRLEED (High Resolution Low Energy Electron Diffraction) and STM (Scanning Tunneling Microscopy).HRLEED provides information in reciprocal space while STM gives real space topographic images of the surface structure. Te was evaporated from a Knudsen cell at a substrate temperature of 200 C. We investigated the effect of coverage and temperature on the Te growth.

EXPERIMENT

The experiments were performed in a standard UHV-chamber at a base pressure of approximately 10^{-10} torr. The chamber is equipped with a HRLEED or SPA-LEED [4,5] and an STM [6] system. Prior to introduction to the vacuum chamber, samples receive an *ex-situ* treatment that is they are first boiled in a NH_3OH solution to remove organic contaminants then submerged in a dilute HF solution to strip off the native oxides; finally, they are boiled in an HCl solution to grow a thin protective oxide layer which is removed upon annealing. *In-situ*, samples are annealed at 1100 C° or until bright diffraction spots are observed. Both STM and HRLEED measurements were performed on Si(100) with misorientation angle ϑ= 0.5°, 1.5° and 8° towards the [110] direction and ϑ= 4 towards the [130] direction. For HRLEED measurements, profiles of the specular beam (along the [011] and [01$\bar{1}$]) at room temperature for different electron energies were recorded by means of an electrostatic deflecting system that scans the electron beam across the aperture of a channeltron detector [5]. By varying the energy of the incident electrons one changes the interference condition which is described by, S, the scattering phase. S is defined as the phase difference between electrons scattered by first and second layer atoms in multiples of the wavelength: integer values of S correspond to an *in-phase* scattering condition while half-integer values correspond to an out-phase scattering condition. STM measurements were also performed on the different Si(100) misorientations at room temperature.

Te was evaporated from an effusion cell, and the deposition rate was monitored by measuring the ion current.

RESULTS

One can gather qualitative and quantitative information from HRLEED. The first information is just a basic visual inspection; the existence of spots implies periodicities of the substrate. The shape of a spot profile provides information on atomic steps and island growth; a splitting indicates a regular step array, a broadening means a random step arrangement etc.... . The second information is extracted (in the case of a broadening) from the equation:

$$\frac{a}{D}=\frac{\Delta K_{//}}{K_{10}} \qquad (1)$$

where a is the lattice spacing, $\Delta K_{//}$ is the full width at half maximum of the broadened beam, K_{10} is the distance in reciprocal space between the specular beam and the (10) beam and D is the average terrace width. In the case of a regular step array the terrace width is simply inversely proportional to the splitting distance [7]. From the energy (or $K_{\perp}$) dependence of the spot profile, the step height can be deduced. For $\vartheta = 0.5°$ and 1.5°, the out-phase profile of the specular beam showed a broadening which was fitted with a Lorentzian then the FWHM was deduced. A model based on the kinematic approximation and random array distribution was developed [8] with γ being the total probability of meeting a step such as:

$$\gamma=\sum_{q=1}^{Q}\gamma_q. \qquad (2)$$

where γ_q is the probability of meeting an atom displaced vertically (either up or down) by qd (q is an integer) and Q is the maximum step height. If the occurrence of a step a one site is independent of its occurrence at another one, then one can assume a geometric distribution for the terrace width given by:

$$P(M)=(1-\gamma)^{(M-1)}\gamma \qquad (3)$$

where the average terrace width is then given by:

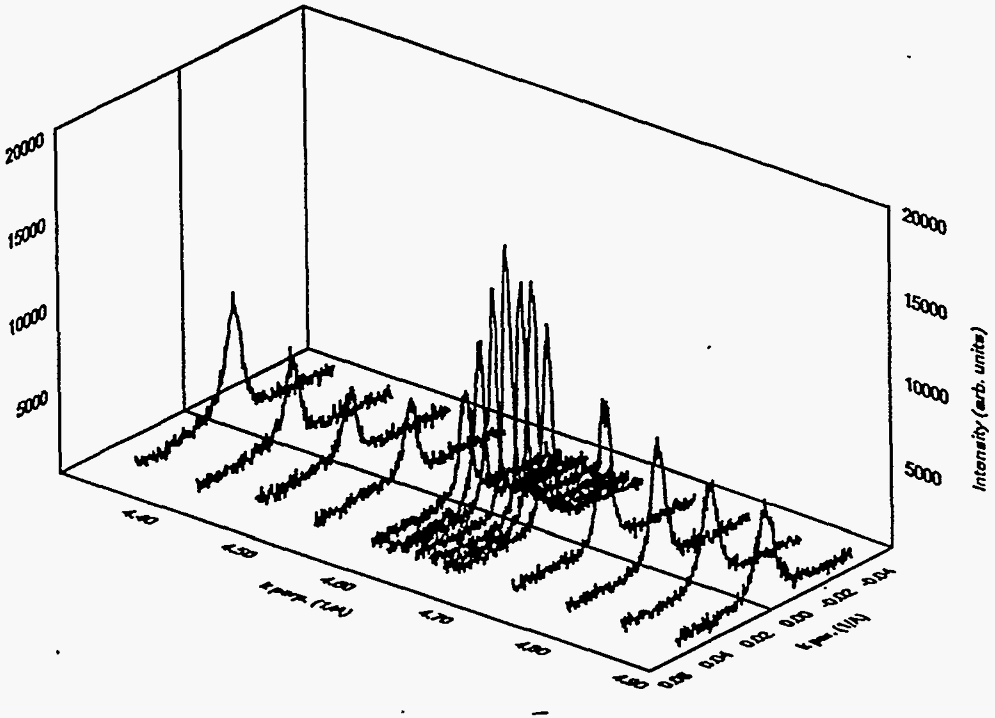
$$\langle L\rangle = \frac{a}{\gamma} \tag{4}$$

In the case of Si(100), the (2×1) surface reconstruction had to be taken into account. For this reason a two dimensional model was considered [9] with γ_1 and γ_2 being respectively the step probabilities in the $\vec{a}_1$ and $\vec{a}_2$ directions ($\vec{a}_1$ and $\vec{a}_2$ are the horizontal unit vectors in the [011] and $[01\bar{1}]$ directions). On neighboring terraces with monatomic step height the two probabilities have to be exchanged because of the (2×1) surface reconstruction. At the out-phase condition, the total intensity is the incoherent addition of the intensities scattered from different terraces. Close to the specular reflection (for $\gamma_1 = \gamma_2$) the resulting intensity profile at out-phase reduces to a Lorentzian along the [011] direction:

$$I(u) = C\left[\frac{(1-\rho^2)}{(1-\rho)^2+\rho u^2}\right] \tag{5}$$

where $\rho = f$, f is called the "boundary structure factor" or average phase factor for pairs of neighboring sites which is a function of the step probability [8].

For $\vartheta = 0.5°$and $1.5°$, the profile of the specular beam changed from intense and narrow at in-phase to weak and broad at out-phase. Figure 1 gives a typical three dimensional measurement (only a selected number of spectra are shown).



1.5 deg. miscut, T=RT, along the $[0\bar{1}\bar{1}]$ direction

Figure 1. In/out of phase intensity profiles for the (00) beam at room temperature (1.5 °)

The broadening was fitted with a Lorentzian and from the FWHM the step probability was deduced. The average terrace width decreases with increasing angle (from 133 A to about

70 A). STM measurements on Si(100) with $\vartheta = 0.5°$ and 1.5° misorientation angle showed monatomic and diatomic steps; the terrace width has a distribution with an average value close to that deduced from HRLEED measurements. For $\vartheta = 8°$, the LEED pattern showed only one domain with a splitting of the spots at out-phase; the terrace width was deduced from the splitting distance [7] and amounted to 18 A, (Figure 2) and the step height was calculated and was found to be diatomic (which is expected since only one single domain was observed in the LEED pattern).STM measurements confirmed the HRLEED result. For $\vartheta = 4°$ misorientation towards $[130]$, the LEED pattern showed a

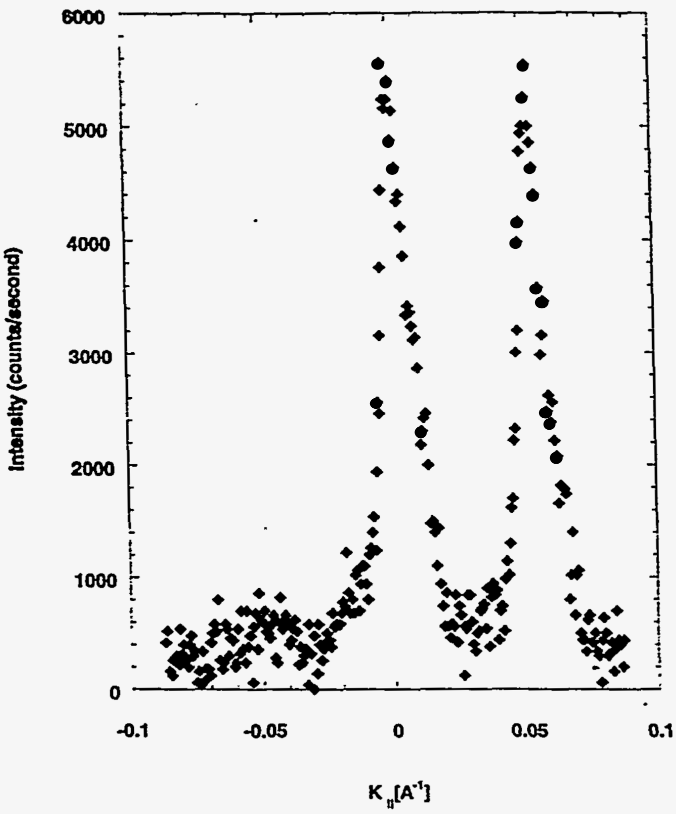


Figure 2. Splitting of the (00) beam at out of phase conditions (8 ^{0}).

splitting (typical to a regular step array) and not only a (2×1) reconstruction in the $[011]$ direction but also an additional weak (2×1) reconstruction in the $[01\bar{1}]$ direction. The splitting was not along one of the principal axes ($[011]$ or $[01\bar{1}]$); this was no surprise since the misorientation is towards [130] (the step edge is neither parallel nor perpendicular to the dimerizational direction). The average step height deduced from the energy dependence of the specular beam profile amounted to d = 2.7 A; the terrace width amouted to 36 A. This suggests a diatomic step height. However, the presence of a weak (2×1) surface reconstruction in the $[01\bar{1}]$ direction leads one to think that the surface is not purely diatomic but the diatomic character overwhelms the monatomic one. An important point that needs to be mentioned is the (2×8) reconstruction on the 1.5° misoriented Si(100); from a previous publication [10], this was attributed to a nickel contamination which would make the surface Si atoms rearrange in such a fashion where single dimers would be missing periodically leading to a (2× 8) surface reconstruction .The question that we are addressing is why in the series of experiments we ran this reconstruction appeared only on the 1.5° misoriented Si(100) even though all the samples were treated the same way. So, if there was a nickel contamination because of the way samples were handled then the (2×8) reconstruction should have been seen in all the other misoriented Si(100) samples but that was not the case. That leads us to one conclusion: the misorientation angle and stress trigger the (2× 8) reconstruction. We also performed HRLEED measurements on the 1.5° misoriented Si(100) sample at T=523 K°; no major change has been noted concerning the terrace width and the step height.

The effect of Te is quite significant. The measurements were performed at 200 C after annealing to 275 C. After this process there is only a monolayer of Te on the surface and the structure changes from 2x1 to 1x1. We observed a decrease in the terrace width for the 0.5 ^{0} misorientation from about 133 A to 17 A. A similar phenomenon is observed for the 1.5 ^{0} misoriented sample, the terrace width is reduced to 18 A. For the 4 ^{0} sample we measured a terrace width of 36 A that is reduced to 16 A when Te is present on the surface. The splitting disappears, indicating no regular array of the terraces. The sample with an 8 ^{0} misorientation retains the structure after 1ML of Te, with diatomic step heigths. The splitting of the (00) spot is present. For low coverages where the surface structure is 2x1 shows a great amount of disorder in terrace widths is shown. More measurements are necessary before we can reach any conclusions about the low coverage samples. We did not obtain very good STM images with the Te samples; this work is still in progress.

CONCLUSION

HRLEED and STM combined represent a powerful tool for defect characterization. While HRLEED gives information on the cleanness and periodicity of the surface atomic arrangement, STM allows direct imaging of the surface. Except for the 8° misorientation in which case a regular step array with diatomic step height was observed, for the other misorientations the terrace width was variable (it justifies our use of the geometric distribution of the terrace width) but its average value was agreed-upon by both STM and HRLEED. The average terrace width decreased with increasing misorientation angle and with Te coverage. Another point is that a mixture of diatomic and monatomic step heights was observed by STM on 0.5° and 1.5° misoriented Si(100) which proves that one can not assume purely monatomic step height for low misorientation angles. Our results do not agree with the belief that at low miscut angle A and B terraces are equal and that as the misorientation angle increases the B terrace tends to be wider than the A terrace. In fact, pairing of terraces was not observed at all. As a matter of fact this just proves that terrace and step distributions are not only affected by the amount of misorientation angle but also by the preparation procedure (in-situ and ex-situ).

ACKNOWLEDGMENT

The authors acknowledge the support of the U.S. DOE BES #W-31-109-ENG-38

REFERENCES

[1] Y. Lo, R.N. Bicknell, T.H. Myers, and J.F. Schetzina, J. Appl. Phys. 54 (1983) 4238
[2] R. Sporken, S. Sivananthan, K.K. Mahavadi, G. Monfroy, M. Boukerche, and J.P. Faurie, Appl. Phys. Lett. 55 (1989) 1879

[3] Y.P. Chen,) Ph.D. Thesis Chicago (1995)
[4] K.D. Gronwald and M. Henzler, Surface Sci. 117 (1982) 180
[5] U. Sheithauer, G. Meyer, and M. Henzler, Surface Sci. 178 (1986) 441
[6] C.J. Chen, Introduction to Scanning Tunneling Microscopy, Oxford Series in Optical and Imaging Sciences
[7] K. Besocke and H. Wagner, Surface Sci. 52 (1975) 653
[8] T.M. Lu and M.G. Lagally, Surface Sci. 120 (1982) 47
[9] S. Heun, J. Falta and M. Henzler, Surface Sci. 243 (1991) 132
[10] H. Niehus, U.K. Kohler, M. Copel,and J.E. Demuth, J. Microscopy, 152 (1988) 735