Extended Abstracts of the 22nd (1990 International) Conference on Solid State Devices and Materials, Sendai, 1990, pp. 1135-1138 **S-F-19**

Step Structure of the Vicinal Si(111) Surface

H. Hibino, Y. Shinoda, Y. Kobayashi, and K. Sugii

NTT Basic Research Laboratories

3-9-11, Midori-cho, Musashino-shi, Tokyo 180 Japan

Analyzing reflection high energy electron diffraction (RHEED) patterns from a vicinal Si(111) surface in closer detail shows that the surface consists of (111) and (331) facets due to step bunching. Multilayer step height is estimated using not only RHEED but also scanning electron microscope (SEM) and transmission electron microscope (TEM). The quantativity of the RHEED analyses can be demonstrated by comparing the results of these estimates. The multilayer step heights are gathered as functions of the misorientation angle and cooling rate after annealing. The multilayer step height is shown to be dependent on both factors. Furthermore the cooling rate is shown to influence the perfection of the (331) facet.

1. INTRODUCTION

Reflection high energy electron diffraction (RHEED) is widely used as an easy method for *in situ* evaluation of a crystal surface structure. For example, the periodicity and direction of the surface structure to the bulk structure can be determined by the symmetry of the diffraction spots, and information on crystal growth can be obtained from the intensity oscillation of the diffraction spot. We tried to uncover new surface structure information by analyzing RHEED patterns in closer detail. In this work, the RHEED pattern features (additional streak, Kikuchi line, and diffraction spot profile), that have not been closely examined previously, are used to quantatively evaluate the step structure of a vicinal Si(111) surface, on which step bunching was reported using low energy electron diffraction (LEED)[1] and scanning tunneling microscope (STM)[2]. In addition to RHEED, SEM and TEM are also used to estimate multilayer step height. The propriety of RHEED analysis is discussed comparing these results. Besides, in order to understand the factors determining the step structure of the vicinal Si(111) surface, the effects of the misorientation angle and cooling rate after annealing on the multilayer step height are also studied.

2. EXPERIMENTAL

Our UHV-chamber was equipped with RHEED optics, a cylindrical mirror analyzer and an electron gun for Auger electron spectroscopy (AES). The instrumental limitations of our RHEED optics, which were estimated using a Si(111) surface with a 7×7 structure, were about 100 Ånormal to the incident direction and 1000 Åparallel direction. Base pressure was about 2×10^{-10} Torr, and pressure was kept below 1×10^{-8} Torr during sample annealing at 1000°C.

Vicinal Si(111) surfaces were misoriented 1°-10° to the [11$\bar{2}$] direction. The samples were cleaned according to the Shiraki method[3]. No impurity peaks were detected in the AES spectrum after this treatment. The samples were heated using radiation from a W filament behind the sample. The sample temperature was measured using an infrared pyrometer. The cooling rate after sample annealing ranged from 7(K/sec) to 0.1(K/sec). Scanning electron microscope (SEM) and transmission electron microscope (TEM) images were taken using a HITACHI S-900 and a JEOL 4000EX, respectively.

3. RHEED PATTERN FEATURES

Three features were observed in the RHEED patterns of the vicinal Si(111) surfaces. The first is the additional spots and streaks observed when the incident electron beam is directed parallel to the step edge. The RHEED pattern is almost the same as for a (111) surface with a 7×7 structure, except that the direction of the RHEED pattern against the shadow edge differs from that of the Si(111) surface. However, additional streaks which cannot be explained by the 7×7 structure are observed. An example of a RHEED pattern for such a vicinal Si(111) surface misoriented 6° is shown in Fig. 1. The additional streak is indicated by the arrow. If only monolayer steps are allowed on the vicinal Si(111) surface misoriented 6°, the width of the (111) terrace is about 30 Å, which is samller than two 7×7 unit cells. Thus, if no correlation is assumed to exist between the 7×7 structures of adjacent terraces, the RHEED pattern for such surface can consist of broad 7×7 spots due to narrow terraces. However, the very sharp 7×7 spots in Fig. 1 proves that the (111) terraces are wider than the instrumental limitation of 100 Ånormal to the incident direction. Furthermore, the additional streaks and spots show the existence of another diffraction surface besides the (111) surface.

The above discussions indicate that there are wide (111) terraces (facets) with 7×7 structures and other facet planes due to step bunching. Assuming that the facet plane is inclined 22° from the (111) surface, the calculated diffraction spot positions on the 0th Laue zone reproduced the experimental data well. This result shows that the facet plane is (331).

The second feature is the splitting of nnn Kikuchi lines when the incident electron beam is directed down the step structure. The RHEED pattern from a vicinal Si(111) surface misoriented 6° is shown in Fig.2, and a pair of 444 Kikuchi lines are indicated by the arrows. The electron beam is refracted at the cryatal surface due to the inner potential. The effect of the refraction is largely dependent on the angle between the electron beam and the cryatal surface. The electrons producing the Kikuchi lines are refracted differently at the (111) terrace and the step bunch. The splitting of the Kikuchi lines in Fig. 2 can be adequately simulated on the assumption that the step bunchs are inclined 22° from the (111) surface.

The third feature is the splitting of all the diffraction spots when the incident electron beam is directed up the step structure. The RHEED pattern from a vicinal Si(111) surface misoriented 6° is shown in Fig. 3. In this figure, each Laue zone appears double, and all the diffraction spots split. Unlike the second feature, the third is ascribed to refraction of the incident electron beam. In Ewald construction, the origin of the reciprocal lattice is the crossing point of the line which is normal to the incident plane and passes the incident point and the dispersion sphere whose radius is the wave number in the crystal. For the incident electron beam directed up the step structure, there is a case in which the electron beam is incident on two surfaces, that is, the (111) terrace and the step bunch, resulting in the two origin points of the reciprocal lattice. Thus the Ewald sphere has two crossing points for one kind of reciprocal rod.

All of the RHEED pattern features indicate step bunching on the vicinal Si(111) surface. This RHEED

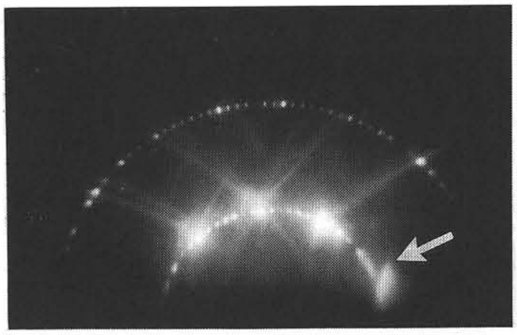

Fig. 1. RHEED pattern from vicinal Si(111) surface misoriented 6°. Incident azimuth is parallel to step edge ([$\bar{1}$10] incidence). Additional streak is indicated by arrow.

Fig. 2. RHEED pattern from vicinal Si(111) surface misoriented 6°. Incident azimuth is 12° from [1$\bar{1}$0] to [11$\bar{2}$]. A pair of 444 Kikuchi lines are indicated by arrows.

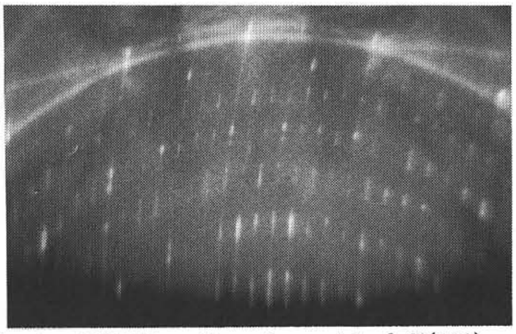

Fig. 3. RHEED pattern from vicinal Si(111) surface misoriented 6°. Incident azimuth is 30° from [1$\bar{1}$0] to [$\bar{1}\bar{1}$2].

observation is consistent with the LEED[1)] and STM[2)] studies. The step bunch is (331) facet at room temperature.

4. ESTIMATION of MULTILAYER STEP HEIGHT

In order to confirm the quantativity of the RHEED pattern analysis, we estimatedmultilayer step height using SEM and TEM images as well as a RHEED pattern. As the analyses using RHEED patterns, the splitting of the diffraction spots is used for a misorientation angle of 1°−2°, and for a misorientation angle of 4°−10° the half width of the diffraction spot is used. The diffraction spots from the regular step array are split due to the periodic terrace structure. From the angular differences $\Delta\theta$ in the splitting spots, step height d can be estimated using the equation below (RHEED spot splitting) .

$$\Delta\theta=(2\pi/kd)\theta_g cos\phi/(\theta_g cos\phi + \theta_c), \quad - \quad (1)$$

where k is wave number, θ_g is glancing angle, θ_c is misorientation angle, and ϕ is azimuthal angle.

We next tried to estimate the multilayer step height from the half width of the diffraction spot (RHEED spot width) . The instrumental limitation is initially obtained as a function of the diffraction angle using the 00 rod of the Si(111) surface, then the half width of the diffraction spot is estimated. (111) terrace width is estimated considering the obstruction of the electron beam by the step. Multilayer step height is estimated from the (111) terrace width and misorientation angle.

Cross-sectional TEM and SEM images were taken *ex situ*. On the sample, Ge cap layers (20-40 Å) were deposited in order to avoid structural change due to oxidation. In the cross-sectional TEM images of the 2° and 4° misoriented surfaces uniformly spaced step structures were directly observed. The cross-sectional TEM image of a vicinal Si(111) surface misoriented 2° is shown in Fig. 4. This figure shows (111) terrace width to be about 600 Å. In the SEM images of the 1°−10° misoriented surfaces, linear contrasts running almost parallel to [1$\bar{1}$0] were observed. These contrasts are ascribed to an increased secondary electron yield when the electron beam is incident on the step edge. The distance between the adjacent contrasts is regarded as one period of the step structure.

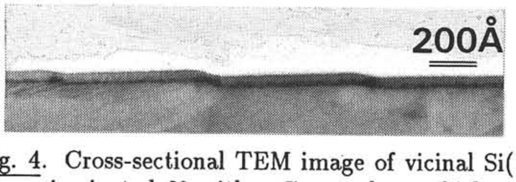

Fig. 4. Cross-sectional TEM image of vicinal Si(111) surafce misoriented 2° with a-Ge cap layer thickness of about 20Å.

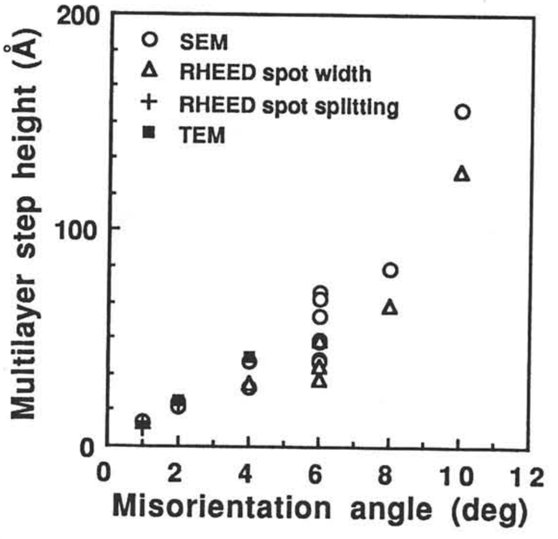

Fig. 5. Misorientation angle dependence of multilayer step height on vicinal Si(111) surface.

Multilayer step height as estimated by the above four methods is shown in Fig. 5. The results obtained using RHEED agree fairly well with those obtained using SEM and TEM. The step heights obtained using the half width of the RHEED spot tend to be smaller than those obtained using SEM and TEM. In the SEM images the terrace width was scattered. This distribution of the terrace widths makes the spot width wider, so terrace width (step height) is apt to be estimated smaller.

Fig. 5 indicates that the larger the misorientation, the higher the step height, and the tendency towards increase is almost linear between 1° and 8°. These results are inconsistent with the result using STM by Swartzentruber et al.[2)], which states that the step bunches on a vicinal Si(111) surface misoriented 1.2−12° consist of about 10 steps (about 30 Å). The first and most important difference between their experimental conditions and ours is in the surface cleaning method. Swartzentruber et al used annealing up to 1250°C. The second difference may possibily be the distribution of the carbon contamination, because the carbon contamination plays the role of a pinning point for step movement. How-

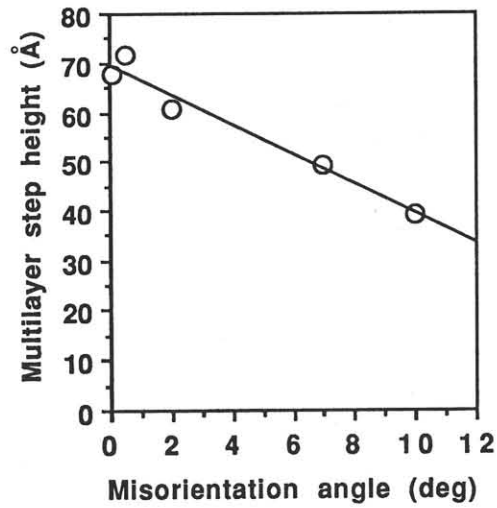

Fig. 6. Cooling rate dependence of multilayer step height on vicinal Si(111) surafces misoriented 6°.

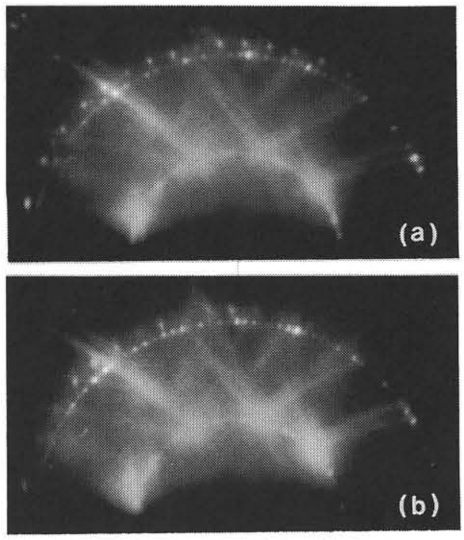

Fig. 7. RHEED patterns from vicinal Si(111) surafces misoriented 10°. (a) cooling rate from 710°C to 550°C is 0.5(K/sec), (b) 6(K/sec).

ever, a carbon signal was not detected in our AES spectrum, and it seems that in our SEM images there is no sign of step pinning. Thus, the effect of carbon contamination on the step structure can be ruled out. The third difference is the sample heating method, which was not addressed in Swaztzentruber's paper. In many cases, the sample is resistively heated, and on a Si(111) surface the direction of the electric current is known to influence distribution of the steps.[4)]

5. EFFECT of COOLING RATE

There are two main effects caused by the cooling rate after sample annealing. One is multilayer step height. The other is the perfection of the (331) facet.

Because kinetics of facetting is an important factor, the size of the step bunch is expected to be dependent on the cooling rate. The cooling rate dependence of multilayer step heights on vicinal Si(111) surfaces misoriented 6° is shown in Fig. 6 for a cooling rate of between 830°C and 700°C. In this figure, the slower the sample is cooled, the higher the step height.

When the misorientation angle is larger than 6°, superstructure spots from the (331) facet were usually observed. Superstrucutre spot intensity is dependent on the perfection of the (331) facet. The RHEED patterns of vicinal Si(111) surfaces misoriented 10° are shown in Fig. 7(a) and (b). The samples in Fig. 7(a) and (b) were cooled at 0.3K/sec and 6K/sec from 710°C to 550°C, respectively. The superstructure spots in Fig. 7(a) are stronger and sharper than those in Fig. 7(b), indicating that the slow cooling improves the perfection of the (331) facet.

6. SUMMARY

Analyzing the RHEED patterns in closer detail confirms that step bunching occurs on a vicinal Si(111) surface, and this surface consists of (111) and (331) facets. Multilayer step height was estimated using not only RHEED, but also SEM and TEM. The multilayer step heights were gathered as a functon of the misorientation angle. It was shown that the multilayer step height is highly dependent on misorientation angle. Multilayer step height is also shown to be dependent on cooling rate, and the cooling rate is an important factor in the perfection of the step bunch structure.

ACKNOWLEGDEMENT

We would like to thank Mrs. S. Ishizawa and Mr. M. Seki for taking the cross-sectional TEM images.

REFERENCES

1) R. J. Phaneuf and E. D. Williams, Phys. Rev. Lett. 58 (1987) 2563.

R. J. Phaneuf and E. D. Williams, and N. C. Bartelt, Phys. Rev. B38 (1988) 1984.

2) B. S. Swartzentruber, Y. W. Mo, M. B. Webb, and M. G. Lagally, J. Vac. Sci. Technol. A7 (1989) 2901.

3) A. Ishizaka and Y. Shiraki, J. Electrochem. Soc. 133 (1986) 666.

4) A. V. Latyshev, A. L. Aseev, A. B. Krasilnikov, and S. I. Stenin, Surf. Sci. 213 (1989) 157.