

**Step Bunching on Si(111)**  
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**Abstract**

In this project Si(111) samples were studied in an Ultra High Vacuum (UHV) system under Direct Current (DC) resistive heating to determine step bunching characteristics for different temperature and mass flux conditions. The goal of the project is to verify the step bunching patterns reported over a range of temperature and flux conditions by other authors as well as to determine some characteristics such as the bunching patterns, in regions that are not well reported. We find the edge of the low regime at  $\sim 950^\circ\text{C}$ , where steps bunch along the current step-down direction under both net sublimation and net growth. The middle regime appears to be  $\sim 1060^\circ\text{C}$  to  $1160^\circ\text{C}$ , where step bunching occurs in the current step-up direction under net sublimation. We find that at  $1060^\circ\text{C}$  under net growth that the bunching pattern does not change to current step-down as reported.

**Introduction**

Silicon is the most commonly studied semiconductor due to its importance in industry and technology. Control of the surface morphology at the level of atomic steps would be extremely advantageous to the creation of nano-scale silicon devices. The step bunching configuration on the surface of the Si(111) crystal has been shown both experimentally [1,2,3] and theoretically [4,5] to depend on the direction of the heating current flowing through the sample as well as the temperature and surface saturation conditions, e.g. net-

sublimation, growth, or equilibrium. Step bunching occurs when the steps on the vicinal surface become unstable under motion and come together. Fig. 1(b) shows surface step rearrangement in the form of a step bunch. Many different theories and mechanisms exist as the reason behind this behavior. Different mechanisms, such as the Ehrlich-Schwoebel

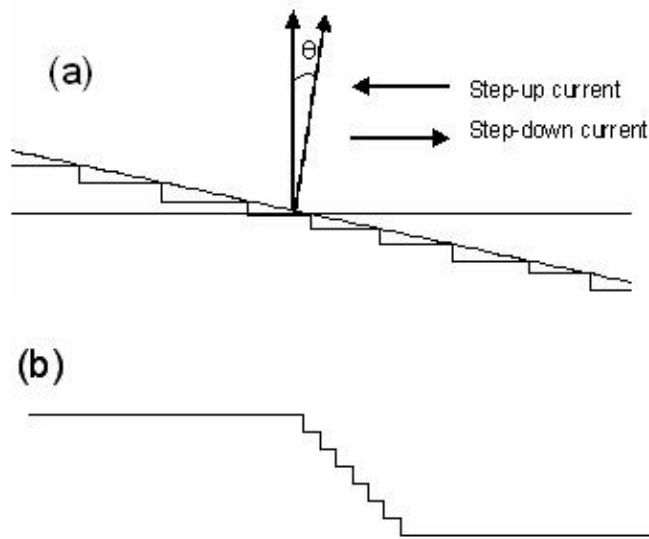


Fig. 1. One dimensional schematic of steps on Si(111) surface. (a) Miscut angle  $\theta$ , arrows give current direction. (b) Steps that have formed a bunch.

Barrier and electromigration seem to dominate under different conditions, making this phenomenon rather interesting and complicated to study. Step bunching during DC heating induced net sublimation has been reported to occur in the step-down current direction in the two temperature regimes of 945-1110°C, 1260-1320°C, and in the

step-up current direction in the range 1160-1240°C. Under net deposition, the step bunching patterns have been reported in one study to remain the same for the low and high regimes, but reverse for the middle regime [3]. The patterns on silicon in the temperatures between these regimes, in particular 1000-1110°C, have not been well documented and part of the goal of this project is to record the behavior of the silicon surface in this “gray areas”.

In addition to the dependence on temperature and current direction, step bunching patterns depend on the net mass flux on the surface. When Si is heated, atoms break

away from the steps and move to the terrace. As they gain more energy, they can break free from the terrace and sublime off the surface. To compensate the evaporation process we can apply a flux using a silicon source (electron beam evaporator) so that the surface on the whole is sublimating more slowly, under equilibrium, or receiving a net flux.

In this work, using optical microscopy and Atomic Force Microscopy (AFM), we characterize the necessary conditions for step bunching in the step-up and step-down current directions on Si(111) samples. Step-up and step-down directions are defined along the direction of the current, and are the result of the sample miscut (see Fig. 1(a)).

In addition to making and studying physical samples, we have made an effort to model the surface through mathematical equations. The basis of this model is the diffusion equation, which is used to describe how atoms on the surface (hereafter referred to as adatoms) flow due to diffusion, attachment/detachment at the step edges, or sublime away into vacuum. Using boundary conditions at the step edges and varying different parameters it is possible gain a better understanding of what is the dominant mass flow mechanism responsible for the morphological changes that occur during the annealing process.

### **Procedure/Experimental Setup**

The experiments were carried out in a UHV system (base pressure  $1 \times 10^{-10}$  torr) equipped with an electron beam (e-beam) evaporator (Tectra, GmbH E-flux model evaporator) and sample load-lock system. To calibrate the ebeam evaporator, silicon samples were placed in vacuum covered by a mask with three slits near the center. Depositions on these samples were carried out for various values of current recorded by a flux

monitoring electrode. This electrode, which is different from the standard quartz crystal monitor that is conventionally used, measures the current of  $\text{Si}^+$  ions flowing out of the evaporator and gives a reading of the amount of silicon which is being deposited. The samples were then removed from vacuum and the thickness of the deposited silicon was

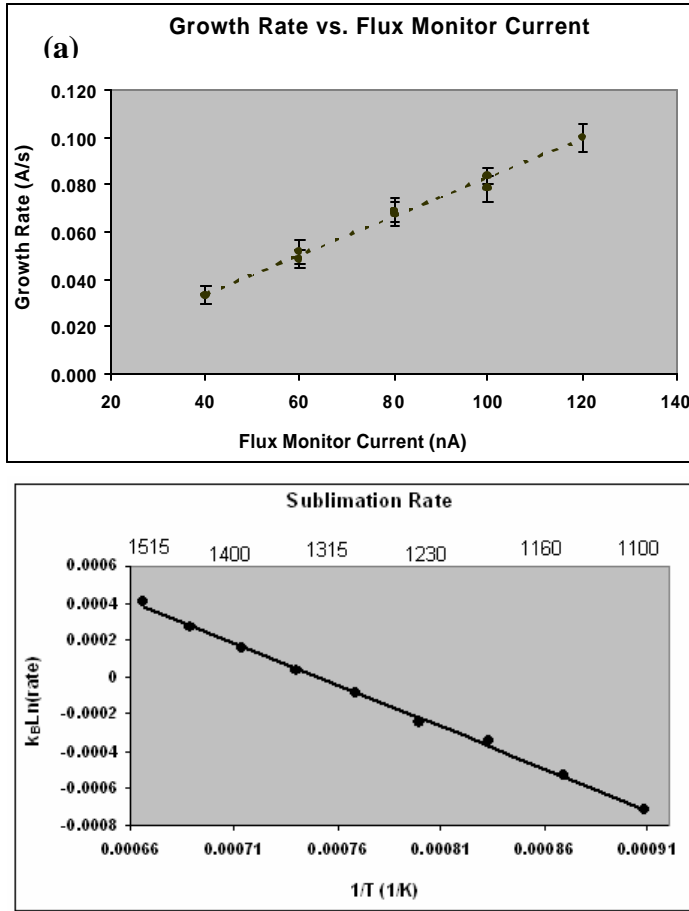


Fig. 2. Graphical data giving (a) the e-beam deposition rate and (b) the sublimation rate from the sample surface. The y-axis units are natural log of the rate in  $\text{\AA}/\text{s}$  times  $k_B$ . Along the top of the x-axis are the temperatures listed in Kelvins.

measured with a surface profilometer (Tencor AS5000 Alpha-Step Profiler). With a thickness and time of deposition measurement, the deposition rate from the evaporator was determined. Fig. 2(a) shows graphical data for the calibrated e-beam evaporator. Using vapor pressure tables, the sublimation rate from the silicon surface was estimated. With these two pieces of data, we were able to determine the required flux monitor current reading necessary to place the sample

under net growth by extrapolating the sublimation rate from the vapor pressure table [6].

Fig. 2(b) shows the vapor pressure data converted to a sublimation rate. Note, our sublimation rate measured at  $1060^\circ\text{C}$  corresponds to a temperature of  $\sim 975^\circ\text{C}$  in the

vapor pressure plot. Therefore, for all of our extrapolations for sublimation rates were scaled to account for this 85°C difference. This was measured by placing a hot sample near a masked sample and measuring the deposition rate. A graduate student, Brian Gibbons did the solid angle calculations to determine what the sublimation rate was from the hot sample. I have not verified these calculations.

The silicon samples were cleaved from n-type (Phosphorus doped) wafers with a resistivity of 0.075 – 0.2  $\Omega$ -cm and a nominal miscut of 0.3 degrees. Typical sample dimensions were 25 mm x 6 mm x 0.4 mm. These pieces were dimpled using a rotating

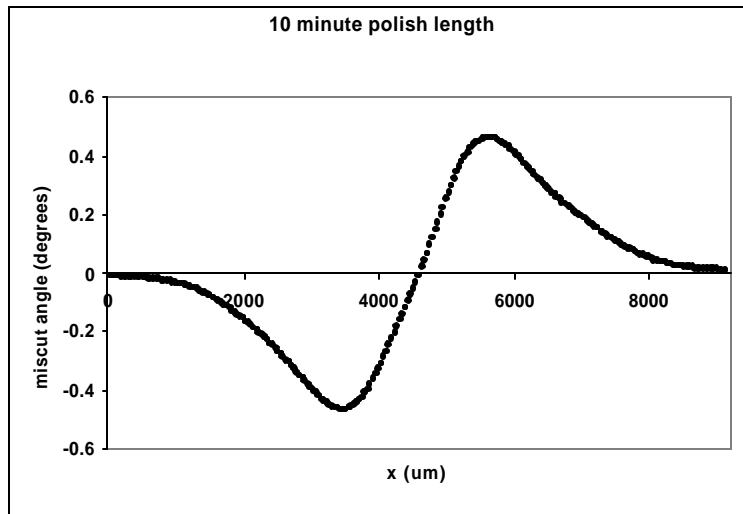


Fig. 3. Miscut angle as a function of position. The center of the dimple is at approximately  $x = 4500\mu\text{m}$  and is where the graph crosses the x-axis.

sample stub and grinding wheel (VCR Dimpler D300). The dimple created a miscut angle on the surface that was great enough to overcome the vicinal miscut angle and

therefore allowed measurement of both step-up

and step-down on the same sample. Fig. 3 shows the miscut angle as a function of the distance from the center of the dimple.

The samples were placed in vacuum and attached to two current leads. Before the high temperature anneal the samples were degassed at  $\sim 740^\circ\text{C}$  for 3 hours.  $740^\circ\text{C}$  was used as an out gassing temperature due to the fact that it is high enough to remove contaminants from the sample, but is just under the temperature required to remove the protective, non-

reactive native oxide layer that naturally forms on the surface of silicon. The pressure in the chamber during degassing remained in the low  $10^{-10}$  torr range. After degassing the samples were repeatedly flashed (30 times) to  $\sim 1150^{\circ}\text{C}$  for a few seconds. A DC voltage was applied with the electric field/current running parallel to the primary wafer flat (Fig. 2(a)) to resistively heat the samples at the desired temperature. Samples were heated to between  $950^{\circ}\text{C}$  and  $1160^{\circ}\text{C}$  with and with out Si flux for 3 – 6 hours. After the annealing

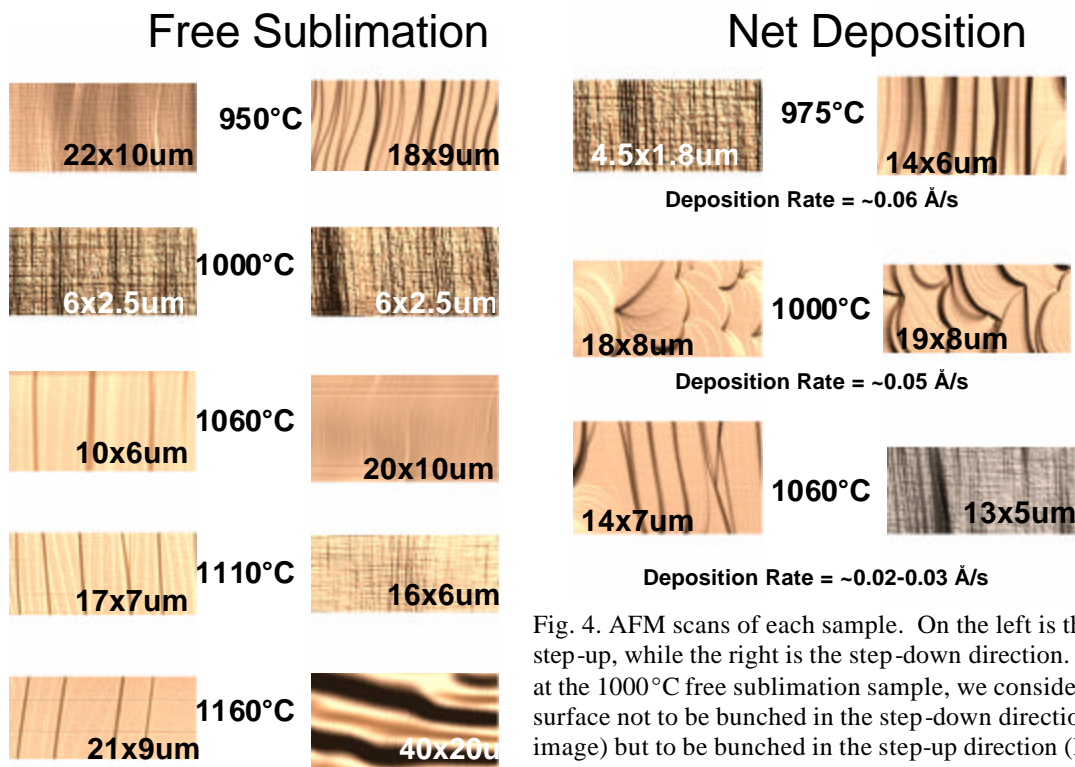


Fig. 4. AFM scans of each sample. On the left is the current step-up, while the right is the step-down direction. Looking at the  $1000^{\circ}\text{C}$  free sublimation sample, we consider the surface not to be bunched in the step-down direction (right image) but to be bunched in the step-up direction (left image).

process, the samples were examined *ex situ* under an optical microscope for general trends and then with an AFM to determine the microscopic surface features.

## Results

We annealed samples under free sublimation at the temperatures of  $950^{\circ}\text{C}$ ,  $1000^{\circ}\text{C}$ ,  $1060^{\circ}\text{C}$ ,  $1110^{\circ}\text{C}$ , and  $1160^{\circ}\text{C}$ . The  $950^{\circ}\text{C}$  sample bunched in the step-down current direction, but not the step-up. We believe this sample to reside in the low temperature

regime. The 1060, 1110, and 1160°C samples all bunched in the step-up current direction, but not in the step-down. This bunching pattern is indicative of the previously reported middle regime. Therefore, we have concluded that the low regime lies  $T = 950^\circ\text{C}$ , whereas the middle is in the range  $1060^\circ\text{C} = T = 1160^\circ\text{C}$ . A higher temperature regime has been reported to exist starting at  $\sim 1200^\circ\text{C}$  where the step bunching pattern resembles that of the low regime—bunching in the current step-down direction, but not step-up. However, this range was not of interest to this study. We also produced a sample annealed at  $1000^\circ\text{C}$ . This sample did not definitively bunch in either current direction. We believe it to fall into the transition zone between the low and middle regimes where bunching in one direction stops and the other starts.

The step bunching behavior for the low and middle regimes seem to match well with previously published works. The actual observed temperatures, however, do not correlate with published numbers. We find that our pyrometer reading is approximately  $100^\circ\text{C}$  lower than other reported temperatures, but still have similar temperature intervals defining the distinct regimes (low, transition, and middle). For example, we find the upper edge of the lower temperature region to be at  $\sim 950^\circ\text{C}$  where Metois *et al.* and Yang *et al.* reported this regime to end at  $1050^\circ\text{C}$ . Similarly these groups report the middle regime to start at  $1160^\circ\text{C}$ , whereas we find it to begin at  $\sim 1060^\circ\text{C}$ .

In addition to free sublimation samples, we annealed several samples under net deposition. These samples were annealed at  $975^\circ\text{C}$ ,  $1000^\circ\text{C}$ , and  $1060^\circ\text{C}$ . The  $975^\circ\text{C}$  sample bunched in the current step-down direction, but not step-up with a deposition rate of  $\sim 0.06 \text{ \AA/s}$ . This suggests that it belongs in the low regime where bunching has been reported not to change regardless of flux conditions. In between the low and middle

temperature regimes, we have found a sample annealed at 1000°C (deposition rate of  $\sim 0.05 \text{ \AA/s}$ ) displays bunching that is symmetric about the dimple. While there was bunching, it appeared to be induced by pinning sites. The suspected source of carbon contamination is the e-beam evaporator and increased chamber pressure during deposition. The 1060°C sample behaved just like the 1060°C free sublimation sample, i.e. no change in the bunching pattern with a net sample flux of  $\sim 0.02\text{-}0.03 \text{ \AA/s}$ . This is interesting as it had been reported that the bunching direction should change in the

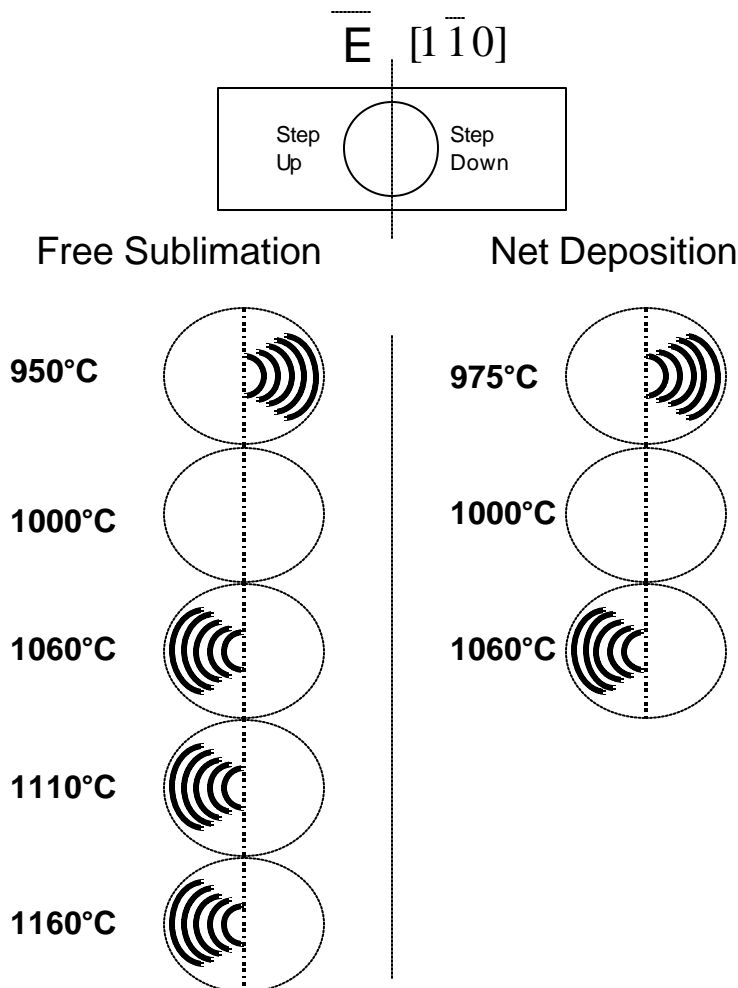


Fig. 5 Summarization of results. Dark bands represent step bunching, clear semi-circles represent no bunching.

middle direction under net growth. More work needs to be done on samples that we believe to be farther into the middle regime (i.e. 1110°C and 1160°C) to verify this result. To determine the affect of pinning sites on the step bunching patterns, we ran a sample under the same conditions as above: 1060°C,  $\sim 0.02\text{-}0.03 \text{ \AA/s}$  deposition rate, except that we used an AC

voltage to eliminate the electromigratory effect. No significant bunching was observed, so we conclude that the surface contamination is not a major source of bunching.

In the future we would like to run a few samples under net growth at temperatures of 950°C, and above 1060°C to check for the appropriate bunching patterns. We would also like to reduce the growth rate so as not to over-saturate the surface. Samples cannot be run too much above 1060°C due to the limiting factor of the e-beam evaporator.

### **Discussion/Conclusion**

In this section we attempt to explain some of the physics behind what causes the step bunching in Si(111). We have looked at two documented mechanisms that have been proposed to contribute to step bunching. It is still unclear whether they are what cause the bunching or how they contribute to what is occurring on the atomic scale.

The first mechanism is what is known as the Ehrlich-Schwoebel Barrier. It is an inequality in the step attachment/detachment coefficient from the two adjacent terraces. Therefore the adatoms on the terraces in front and behind the step have an unequal probability of attaching to the step. Instead of then looking at the adatom concentrations on both sides of the step the same, there is some prefactor attached to each. It has been calculated that the attachment coefficient on the leading step side may be only ~20% of that on the trailing side. This asymmetry may be enough to induce step bunching under some situations.

The second mechanism is Surface electromigration. Surface electromigration is the motion of mass caused by an electric field. This field is correlated with the DC voltage that we applied on our samples to induce step bunching. While this electromigratory effect alone is not enough to induce bunching, it is likely that this effect may “turn on”

some effects depending on temperature. This may be enough to cancel out the effect in one direction, and amplify it in the other (as the step train switches from current step-up to step-down through the middle of the dimple). One of the effects that may be coupled with surface electromigration is permeable or transparent steps. Under this condition, adatoms are allowed to hop over steps without attaching to the edge. This effectively allows each step to “see” further than the adjacent terraces and use step bunches as a large source of adatoms, as opposed to only being affected by the size of the adjacent terraces. Another coupled effect is the dependence of the step velocity only upon the size of the terraces adjacent to it. This dependence, however, need not be equal. In the case where the velocity depends more heavily on the leading step than the trailing step (under net sublimation, steps flow away from the center of the dimple so the leading step would be away from the dimple—for net deposition the opposite is true), steps tend to be stable against bunching. However, in the case where the dependence is stronger with the trailing step, small perturbations in the even spacing of steps can become greatly exaggerated and lead to multiple step bunching. This can be found by solving for the equilibrium position of each of step an infinite one-dimensional train of steps [7].

Future plans include designing experiments to verify where and to what extent the domain of each of these effects lies.

## **Conclusion**

We have shown that under net sublimation the low, transition, and middle temperature regimes exist at 950°C, 1000°C, and 1060-1150°C respectively. We found bunching in the current step-down direction at 950°C, no bunching at 1000°C, and bunching in the current step-up direction from 1060-1150°C. Under net deposition the transition regime

exists from 975-1000°C, while the middle regime begins at ~1060°C. We found no conclusive bunching in the regime 975-1000°C, and found bunching in the current step-up direction only at 1060°C. In addition, we tested and calibrated the e-beam evaporator. This now prepares us to run more complex future experiments with a high degree of control.

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### **References**

- [1] Y. Homma, R. McClelland, H. Hibino, *Jpn. J. Appl. Phys.* **29** (1990) L2254—L2256
- [2] Y.-N. Yang, E. Fu, E. Williams, *Surf. Sci.* **356** (1996) pp 101-111
- [3] J.J. Metois, S. Stoyanov, *Surf. Sci.* **440** (1999) pp 407-419
- [4] S. Stoyanov, *Jpn. J. Appl. Phys.* **30** (1991) pp 1-6
- [5] S. Stoyanov, H. Nakahara, M. Ichikawa, *Jpn. J. Appl. Phys.* **33** (1994) pp 254-259
- [6] J. O'Hanlon: *A User's Guide to Vacuum Technology*. (New York, United States, 1980) p. 372.
- [7] P. Bennema and G. Gilmer: *Crystal Growth: An Introduction*, ed. P. Hartman (North Holland, Amsterdam, 1973) pp 317-320.