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# Spatially Controlled Formation of Atomically Flat Si(001) Surface by Annealing with a Direct Current in UHV

Atsushi ANDO, Kunihiro SAKAMOTO, Kazushi MIKI, Kazuhiko MATSUMOTO and Tunenori SAKAMOTO

*Electrotechnical Laboratory (ETL), 1-1-4 Umezono, Tsukuba, Ibaraki 305, Japan*

A technique that the spatially controlled large terraces are formed by barrier structures of step movement such as artificial step bands is proposed. This technique enables to fabricate an atomically flat large Si(001) surface at a controlled position on a wafer by annealing the patterned substrate with a direct current in ultrahigh vacuum (UHV). Single atomic round Si(001) planes of about 4 μm in diameter have been obtained by annealing the 6 μm square patterned substrate at 1000°C for 2 hours.

## 1. Introduction

Silicon is one of major semiconductor materials which is widely used for many kinds of electronic devices. Realizing an atomically flat Si(001) surface in large area is necessary to fabricate nano-structural electronic devices using the Si ULSI technologies without degradation of their performance.[1] It has been reported that atomic steps on Si(001) surface easily move on the surface during annealing in ultrahigh vacuum (UHV) and surface topography changes to a stable state according to the annealing condition.[2-9] When Si(001) surface is annealed at 1000°C by passing a direct current, atomic steps tend to be gathered and to form step bands which separated large terraces of a single atomic plane.[2, 3, 5, 10, 11] The widths of these terraces are a few microns. Although the terraces are an attractive platform for nano-structural electronic devices such as an atomic-step-free MOS device, the positions of the terraces are not controlled. In order to realize nano-structural electronic devices, controlled formation of the terraces at expected positions is necessary.

It is known that step movement is depressed by surface contaminations such as SiC, which results in step bunching. Making use of this phenomenon is interesting for spatial control of large terraces. We propose a technique that the formation of the terraces is spatially controlled by barrier structures of step movement. Artificial step bands, different materials such as silicon oxide and silicon nitride, and defects are available for the barrier structures. These barrier structures are able to be formed on Si(001) surface with expected patterns by a typical lithographic method and this technique is suitable for a device fabrication process.

In this paper, we introduce artificial step bands as barrier structures to depress step movement and show spatially controlled formation of atomically flat Si(001) surface by annealing with a direct current in UHV.

## 2. Experimental

A well-oriented Si(001) substrate (Shin-Etsu Semiconductors Co., Ltd.) was used for a specimen. Its surface normal was off from the [001] orientation by 0 - 7' along the [110] direction and by 3 - 7' along the [$\bar{1}$10] direction. Artificial step bands with depths of 20 - 500 nm were formed into square patterns along [110] and [1$\bar{1}$0] directions by chemical etching. After degreasing with acetone in an ultrasonic bath, the specimens were boiled in a $H_2SO_4$:$H_2O_2$:$H_2O$ mixture (6:1:3 weight ratio) at 100°C for 10 min. To form thin chemical oxide layers on the surface, the specimens were dipped in a 50 wt% HF solution for 30 s and boiled again in the above mixture at 100°C for 2 min. After undergoing these treatments, all specimens were loaded into a UHV system, which base pressure is about 2 x $10^{-8}$ Pa, and were degassed at about 600°C for 8 hours. Surface cleaning by 3 times flashing at around 1200°C for several seconds was performed to remove the chemical oxide layers. To obtain large terraces of a single atomic plane, the specimens were annealed at 1000°C by passing a direct current toward to the [100] direction for 2 hours. The pressure of the system was maintained in the order of $10^{-8}$ Pa during the annealing. The temperature was measured by a pyrometer.

Surface topography was observed with an atmospheric atomic force microscopy (AFM) just after unloading the specimens. AFM experiment was performed using a commercial one (SPA300, Seiko Instruments Inc.). All AFM images shown in this study are topographic ones. Au coated cantilevers with a microfabricated silicon nitride tip (Olympus Opt. Co., Ltd.) were used for AFM observation. Typical values for curvature of tip apex and spring constant were 15 nm and about 0.1 N/m, respectively. The reference force during AFM observation was maintained in the order of $10^{-9}$ N.

## 3. Results and Discussion

We first observed surface topography of unpatterned specimens after the annealing. Figure 1 shows a typical AFM image of unpatterned Si(001) surface after annealing with a direct current toward the [100] direction at 1000°C for 2 hours in UHV. The scanning area of the image is 8 x 8 $\mu m^2$. The step bands run in the [1$\bar{1}$0] direction and intervals of them are about 3.5 μm. The heights of the step bands are about 5 - 8 nm. There are large terraces with an area of a few square microns, which are separated by the step bands in the [110] direction and by double atomic

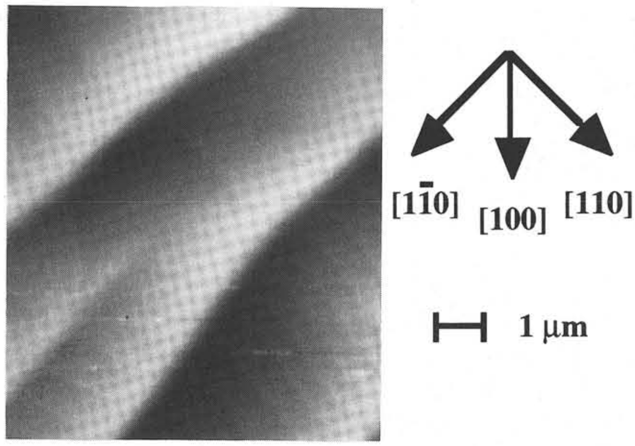


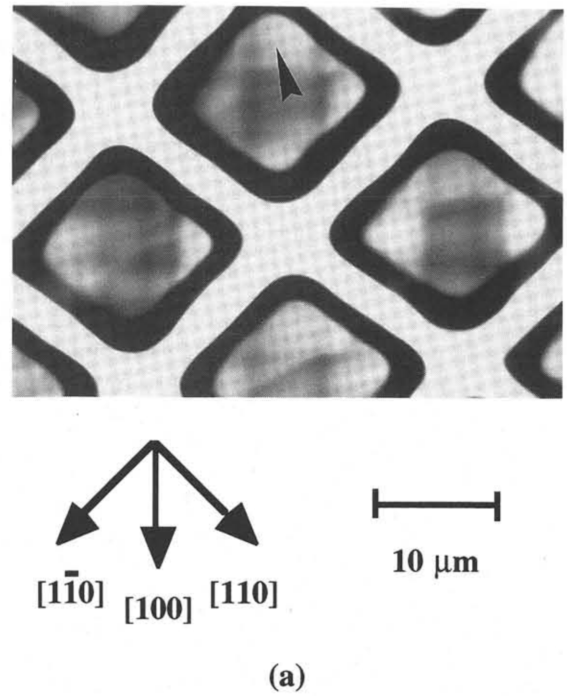

Fig. 1. 8 x 8 μm$^2$ AFM image of unpatterned Si(001) surface after annealing by a direct current toward the [100] direction at 1000°C for 2 hours.

steps in the [1$\bar{1}$0] direction. Although these terraces are large enough for device fabrication, their size and formation position are not controlled as mentioned above.

When square patterned artificial step bands were used as barrier structures, spatial control of the terrace formation was obtained. Figure 2 (a) shows a typical AFM image of 10 μm square patterned Si(001) surface after annealing with a direct current toward the [100] direction at 1000°C for 2 hours in UHV. The scanning area of the image is 40 x 35 μm$^2$. A large flat terrace is formed at the upper corner of each square pattern, which is surrounded by artificial step band and a self-formed step band. A few step bands are observed in the patterned area and their intervals are 2 - 5 μm. Figure 2 (b) shows a magnified AFM image of the large flat terrace indicated by an arrow in Fig. 2 (a) on an atomic step resolution. A single atomic plane with an area about 12.5 μm$^2$ is observed.

Although the spatial control is obtained, the single atomic plane is a small part of the patterned area. It is caused by self-formed step band as shown in Figs. 2 (a) and (b). On the annealing condition (1000°C, 2 hours), step bands are formed at intervals of about 3.5 μm as observed in Fig. 1. A diagonal length of the square pattern is 14 μm and is about 4 times as long as the interval. To fill most of the patterned area with a single atomic plane, it is necessary that the intervals of self-formed step bands are close to a diagonal length of the pattern. In this case, making use of smaller square pattern or extending the intervals of self-formed step bands is needed. Although a longer time or a higher temperature annealing seems to be possible to extending the intervals, there is a fear that the artificial step bands as barrier structures may be destroyed during these annealing. These annealing conditions are not suitable in this case. Then we used smaller square pattern with 6 μm in size, while the annealing condition was the same.

Figure 3 (a) shows a typical AFM image of 6 μm square patterned Si(001) surface after annealing with a direct current toward the [100] direction at 1000°C for 2 hours in



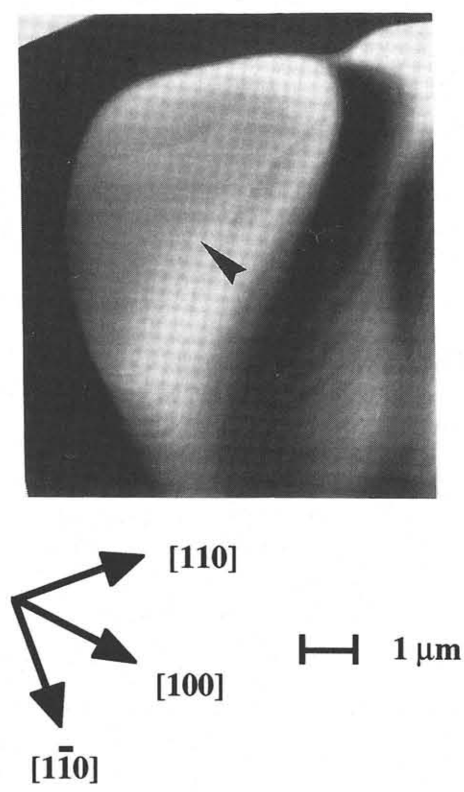




Fig. 2. (a) 40 x 35 μm$^2$ AFM image of 10 μm square patterned Si(001) surface after annealing by a direct current toward the [100] direction at 1000°C for 2 hours.
(b) Magnified image of the square patterned area indicated by an arrow in Fig. 2 (a). An arrow indicates a single atomic plane with an area about 12.5 μm$^2$.

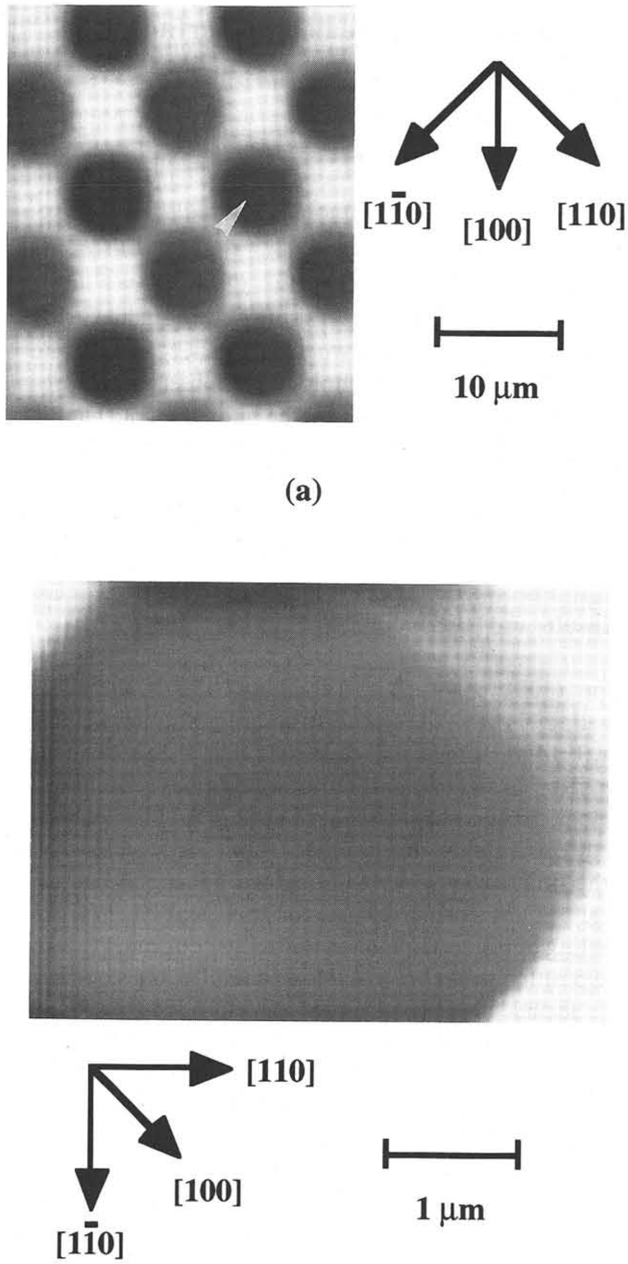



Fig. 3. (a) 30 x 30 $\mu m^2$ AFM image of 6 μm square patterned Si(001) surface after annealing by a direct current toward the [100] direction at 1000°C for 2 hours.
(b) Magnified image of the square patterned area indicated by an arrow in Fig. 3 (a).

UHV. The scanning area of the image is 30 x 30 $\mu m^2$. No step bands are observed in the patterned area. Figure 3 (b) shows a magnified AFM image of the area indicated by an arrow in Fig. 3 (a). Single atomic steps are gathered at the edge of the pattern and most of the area is a single atomic round plane of about 4 μm in diameter. This spatially well-controlled single atomic plane is one of ideal substrates for nano-structural electric device fabrication.

## 4. Conclusion

We used artificial step bands as barrier structures to depress step movement and have successfully fabricate an atomically flat large Si(001) surface at a controlled position on a wafer by annealing the square patterned substrate with a direct current in UHV. With a 6 μm square pattern, most of the patterned area consists of a single atomic round plane of about 4 μm in diameter after the annealing at 1000°C for 2 hours.

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