

Local Oxidation Characteristics of Single Crystal Silicon with an Atomic Force Microscope

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ABSTRACT

In order to investigate local oxidation characteristics on Si (100) surfaces, the local oxidation processing has been carried out by using an atomic force microscope with a Si tip coated with Pt. It has been clarified that the bias voltage where an oxide feature can be generated has a threshold. The threshold voltage is 5~6 V for Si (100) surfaces with a native surface oxide layer. As the bias voltage exceeds the threshold, dimensions of the oxide feature increase with an increase of the bias voltage. Particularly, the height of generated oxide is sensitive to a variation of the bias voltage. In addition, it is demonstrated that the fine crisscross oxide lines with nine intersections ($1500 \times 1500 \times 2.6 \text{ nm}^3$) are successfully fabricated by the local oxidation processing. The height of the intersection is about 0.6 nm higher than that of oxide lines. It indicates that an oxide feature is newly formed on the oxide line generated previously.

Keywords: Anodization, Atomic force microscope, Single crystal silicon, Process characteristics

1 INTRODUCTION

Recently, surface modification with a scanning probe microscope (SPM) has attracted special interest as one of the surface processing techniques in a nanometer scale and investigated vigorously [1]. The nanoscratching with an atomic force microscope (AFM) that removes a material in a nanometer scale is a representative technique [2]. In addition, the generation of nanometer scale oxide by utilizing electrochemical or tribochemical reactions on the surface has been made an attempt and it has been reported that the generated oxide can be used as a mask in the etching [3,4].

In this study, we take notice of the electrochemical local oxidation processing with an AFM as one of the surface processing techniques. In order to investigate and discuss the processing characteristics, a series of local oxidation processing experiments on polished Si (100) surfaces has been conducted by using an AFM with a Si tip coated with Pt.

2 EXPERIMENTAL PROCEDURE

As shown in Figure 1, a water column is formed around

the tip-sample junction in the cause of capillarity when a tip approaches a sample surface closely. Polarizing the sample surface anodically, anions in the water column migrate to the sample surface. They react with the sample surface to form an oxide at the point beneath the tip. A principle of local oxidation using a SPM is summarized as mentioned above.

In this study, oxide features were fabricated on a single crystal Si (100) surface by using the AFM in air. When a bias voltage was applied to the tip coated with Pt, an oxide feature was generated at just below the tip, as shown in Figure 2. The conical Si tip had a radius of curvature less than 60 nm. The Si surface was cleaned in ethyl alcohol by means of ultrasonic waves for 15 minutes. Figure 3 (a) and (b) show processing methods of oxide dots and oxide lines, respectively. In the processing of oxide dots, the tip was held for 10 s applying a DC bias voltage in the range of 2~10 V, as shown in Figure 3 (a). In the processing of oxide lines, the tip was scanned along the [110] direction at a speed of 0.1 $\mu\text{m/s}$ applying a DC bias voltage in the range

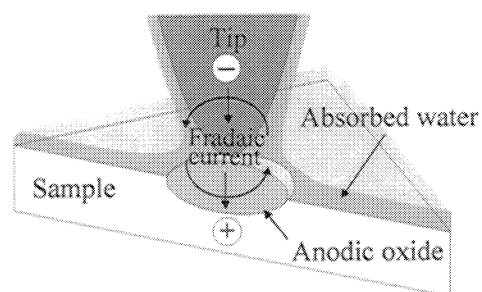


Figure 1: Principle of the local oxidation using SPM.

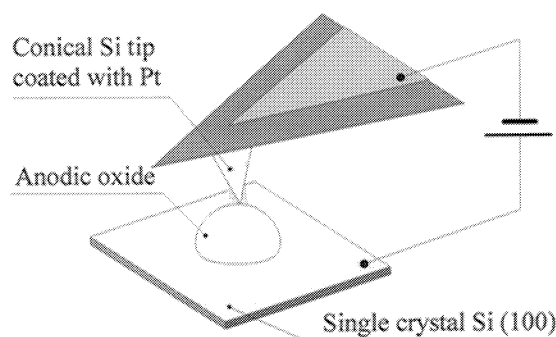


Figure 2: Schematic of the local oxidation processing using an AFM tip.

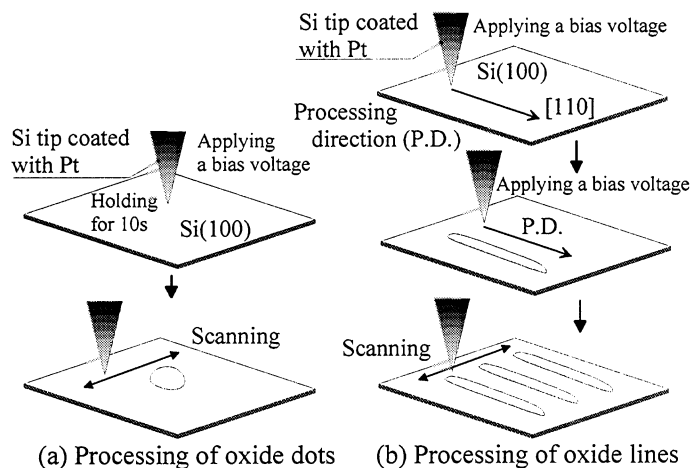


Figure 3: Processing methods of oxide features.

Table 1: Experimental conditions for processing oxide features.

Tool	Si tip coated with Pt
Sample	Si (100)
Bias voltage	1 ~ 10 V
Contact force	10 nN
Applied time in dot processing	10 s
Processing speed in line processing	0.1 $\mu\text{m/s}$
Atmosphere	In air
Humidity	30 ~ 50 %

of 1~10 V, as shown in Figure 3 (b). All experiments were carried out at contact force of 10 nN in humidity range of 30~50%. Experimental conditions for processing oxide features are shown in Table 1.

After processing, the modified surface was immediately imaged using the same tip.

3 RESULTS AND DISCUSSION

3.1 Processing of Oxide Dots

A typical AFM image of an oxide dot processed at a bias voltage of 10 V is shown in Figure 4 (a) and the surface profile curves at sections AA' and BB' are shown in Figure 4 (b). The horizontal sections of the generated oxide were nearly circular and the center of the oxide dot was highest. As the distance between the tip and the Si surface becomes short, the electric field on the Si surface becomes strong. Therefore, it is considered that the center of generated oxide becomes highest. At the bias voltage of 10 V, a height at the center of oxide dot was about 2.5 nm and a diameter at the bottom of oxide dot was about 200 nm.

From the AFM observations, a relationship between the height at the center of oxide dot H_d and the bias voltage E was measured, as shown in Figure 5. In the same way, a relationship between the diameter at the bottom of oxide dot D_d and the bias voltage E was measured, as shown in the same figure. It is found from these results that the oxide

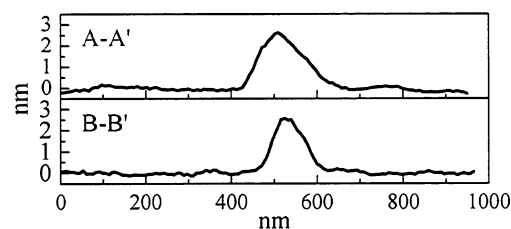
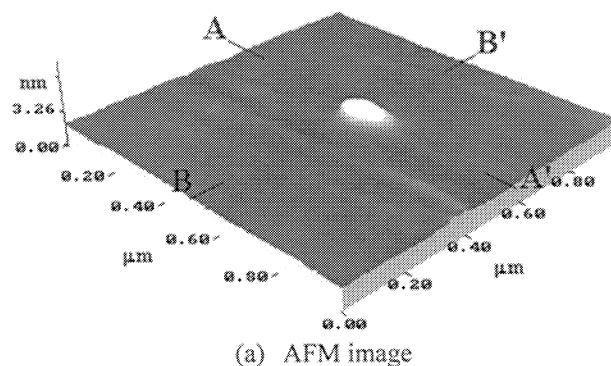


Figure 4: A typical AFM image of the oxide dot processed at bias voltage $E = 10$ V.

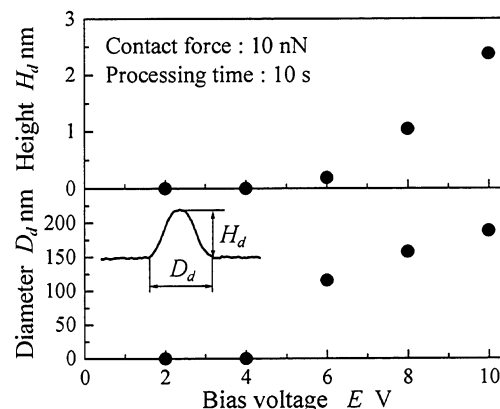


Figure 5: Effects of bias voltage E on height H_d and diameter D_d of the oxide dot.

dot is not generated below the bias voltage of 4 V. This fact indicates that the bias voltage where an oxide dot can be generated has a threshold. This is considered to be caused by a native surface oxide layer. As the bias voltage exceeds 6 V, the height H_d increases rapidly with an increase of the bias voltage. It is the cause that an electrochemical interaction is promoted by the increase in the bias voltage. The diameter D_d is already large at the bias voltage of 6V and increases gently with an increase of the bias voltage. It seems that factors other than the bias voltage, such as a tip radius and a size of the water column, affect the diameter D_d strongly.

3.2 Processing of Oxide Lines

A typical AFM image of oxide lines processed at the

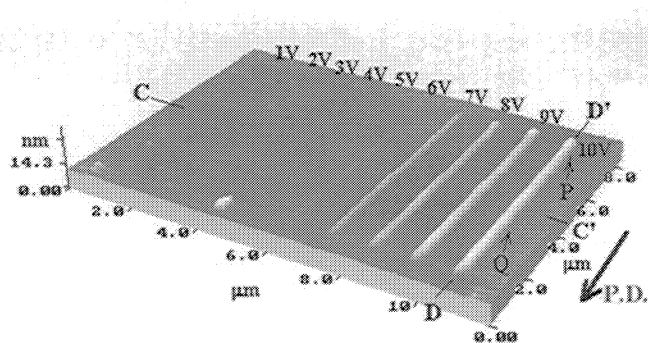
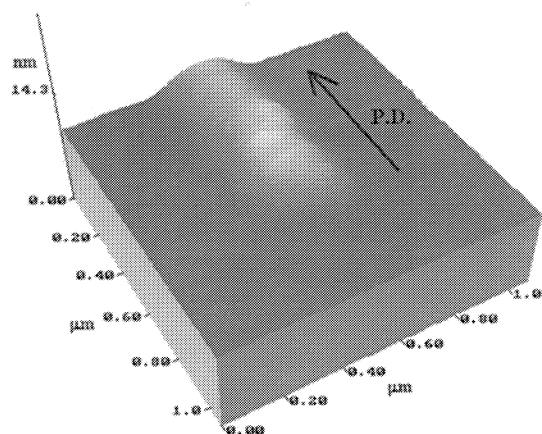
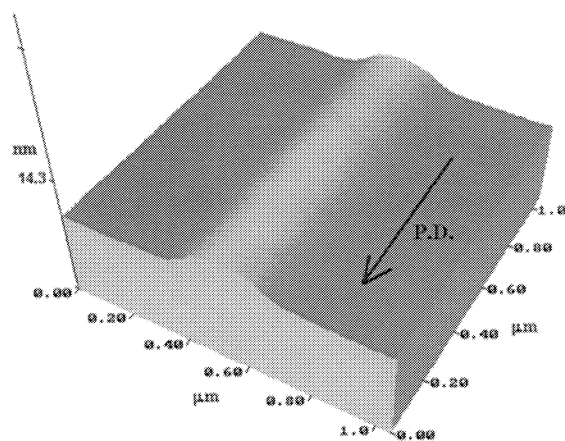


Figure 6: A typical AFM image of the oxide lines processed at bias voltage $E = 1 \sim 10$ V.



(a) Portion P



(b) Portion Q

Figure 7: High magnification AFM images of the oxide line processed at bias voltage $E = 10$ V (Portions P and Q shown in Figure 6, respectively).

bias voltage range of 1~10 V is shown in Figure 6. The oxide line was generated at the bias voltage range of 6~10 V and it was the same as the oxide dot processing. Figure 7 (a) and (b) show high magnification AFM images of the oxide line processed at a bias voltage of 10 V (portions P and Q shown in Figure 6, respectively). As shown in Figure 7 (a), the shape at a process starting point is smooth and roundish although some irregularities are seen. A cross

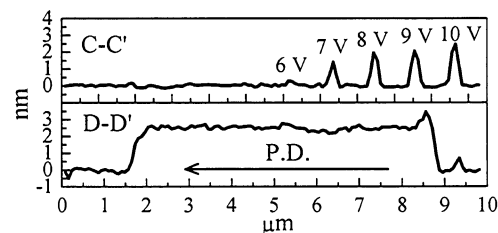


Figure 8: Surface profile curves at sections CC' and DD' in Figure 6.

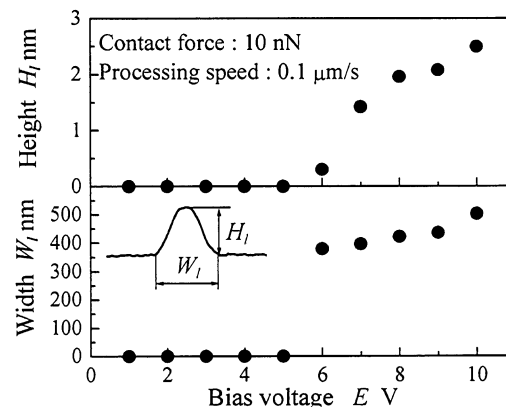


Figure 9: Effects of bias voltage E on height H_l and width W_l of the oxide line.

section of the oxide line has a shape of quite smooth arch, as shown in Figure 7 (b). The surface profile curves at sections CC' and DD' are shown in Figure 8. As the bias voltage exceeds 6 V, a height of generated oxide increases with an increase of the bias voltage. The peak-to-valley roughness of oxide surface along the processing direction is about 0.25 nm.

Figure 9 shows relationships between the height at the center of oxide line H_l , the width at the bottom of oxide line W_l and the bias voltage E . The height H_l is almost equal to the height of the oxide dot H_d processed at the same bias voltage. On the other hand, the width W_l is twice larger than the diameter of the oxide dot D_d processed at the same bias voltage. It is thought that a change in the electric field by moving the tip is the cause of the change in the width of generated oxide.

Figure 10 shows a processing mechanism of the oxide line. When a tip is moved applying a bias voltage, an oxide feature is continuously generated around the tip-sample junction. Because an oxide generating effect is strongest at the tip-sample junction, a cross section of the generated oxide line becomes a smooth arch shape. The same effect acts to a moving direction of the tip. Therefore, it is considered that both ends of the oxide line become smooth and roundish.

3.3 Fabrication of Crisscross Oxide Lines

We have tried to fabricate fine crisscross oxide lines on the basis of the experimental results mentioned above.

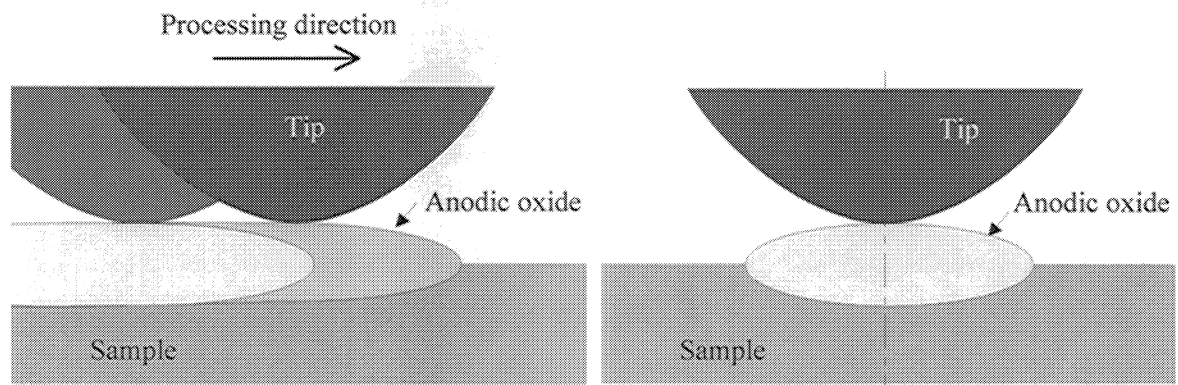


Figure 10: Schematic illustration of the mechanism for processing an oxide line.

Figure 11 shows a typical AFM image of the crisscross oxide lines with nine intersections. The lines were processed in the order, a→b→c→d→e→f, as shown in the figure. Then the crisscross oxide lines are fabricated in the size of $1500 \times 1500 \times 2.6 \text{ nm}^3$. The height at the intersections of the crisscross lines is about 0.6 nm higher than that of oxide lines. It indicates that an oxide feature is newly formed on the oxide line generated previously. However, the height of the newly formed oxide at the intersection is lower than that of oxide generated on Si surface.

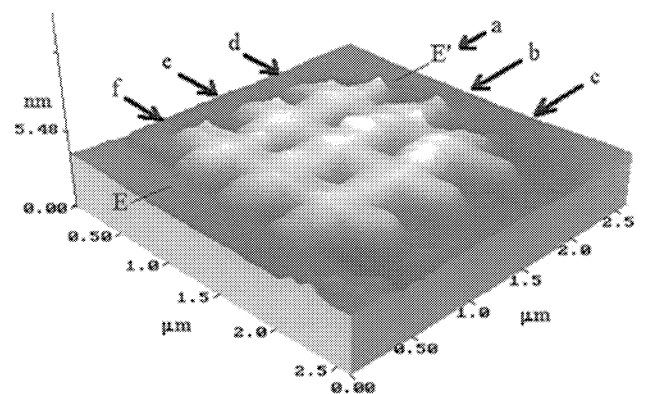
4 CONCLUSIONS

Local oxidation processing experiments on polished Si (100) surfaces have been conducted in air by using an atomic force microscope with a Si tip coated with Pt. Oxide dots and oxide lines have been successfully fabricated and local oxidation characteristics of single crystal Si have been investigated. The main results are summarized as follows:

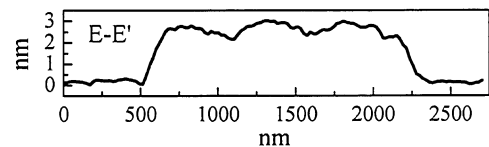
- (1) The bias voltage where an oxide feature can be generated has a threshold on Si surface with a native oxide layer and the threshold voltage is 5~6 V.
- (2) As the bias voltage exceeds the threshold, dimensions of the oxide feature increase with an increase of the bias voltage. Particularly, the height of generated oxide is sensitive to a variation of the bias voltage.
- (3) Fine crisscross oxide lines with nine intersections ($1500 \times 1500 \times 2.6 \text{ nm}^3$) are successfully fabricated by the local oxidation processing.
- (4) At the intersections, an oxide feature is newly formed on the oxide line generated previously. The height of the newly formed oxide is lower than that of oxide generated on Si surface.

REFERENCES

[1] E. S. Snow and P. M. Campbell, "Fabrication of Si Nanostructures with an Atomic Force Microscope," *Appl. Phys. Lett.*, 64, 1932, 1993.



(a) AFM image



(b) Surface profile curve at section EE'

Figure 11: A typical AFM image of fine crisscross oxide lines.

[2] Y. Ichida, R. Sato and K. Takahashi, "Nanometer-Scale Machining of Single-Crystal Silicon Using an Atomic Force Microscope," *Precision Science and Technology for Perfect Surfaces*, JSPE Publication Series No.3, 677, 1999.

[3] H. Sugimura and N. Nakagiri, "Chemical Approach to Nanofabrication: Modifications of Silicon Surfaces Patterned by Scanning Probe Anodization," *Jpn. J. Appl. Phys.*, 34, 3406, 1995.

[4] R. Garcia, M. Calleja and H. Rohrer, "Patterning of Silicon Surfaces with Noncontact Atomic Force Microscopy: Field-induced Formation of Nanometer-size Water Bridges," *J. Appl. Phys.*, 86, 1898, 1999.