# Growth Control of Nano-Needle on Silicon Surface Using Scanning Tunneling Microscope

Seiji Heike, Tomihiro Hashizume and Yasuo Wada

Advanced Research Laboratory, Hitachi, Ltd., 2520 Akanuma, Hatoyama, Saitama 350-03, Japan

Nano-needles are formed on the Si(111) surface when negative ramp voltages are applied to a scanning tunneling microscope (STM) tip. These nano-needles allow the direct imaging of the STM tip, because of their extreme sharpness with an estimated diameter of approximately 2 nm and a maximum height of 20 nm. In this paper, voltage, time, and current dependences of nano-needle growth are examined. Based on the experimental results, we propose that the nano-needle formation mechanism is: Si atom extraction from the Si surface to the tip due to the applied high voltage, migration of the atoms to the tip apex, and redeposition from the tip apex to the sample surface.

#### **1. INTRODUCTION**

We have reported nano-needle formation on Si(111) surfaces and its application to *in situ* tip observation by counter imaging (Needle Formation and Tip Imaging: NFTI)<sup>1)</sup>. The nano-needle was formed by applying negative high voltage to the tip with a current constant, and can also be grown on the tip apex by using positive high voltage. The nano-needle scans the STM tip, and the STM image is obtained as a convolution of the tip apex shape and the nano-needle shape. Since the nano-needle structure is one or two orders of magnitude smaller than the tip, with approximately 2 nm in diameter and 10 nm in height, the convolution image in practice represents the tip apex structure.

This paper proposes a nano-needle formation mechanism to claim the reproducibility and reliability of this technique. The voltage, time, and current dependences of the needle growth height were investigated, and the area with extracted Si atoms of the sample surface were evaluated. In addition, STM images observed after various voltage applications were compared. The needle growth process was analyzed based on these experimental results.

### 2. EXPERIMENTAL

The STM used in this experiment was a homemade ultra high vacuum system with base pressure below  $7 \times 10^{-11}$  Torr. The samples were cut from n-type 0.005- $\Omega$ cm (111) oriented silicon wafers. The STM tips were prepared by electrochemically sharpening tungsten wires and heating them by electron bombardment. The nano needle structures were formed by slowly increasing the tip bias voltage up to around -4 to -10V and kept for about 1 to 60s on a Si(111) surface, while maintaining the tunneling current at 0.2nA. The needle heights were measured as functions of applied voltage, duration time and tunnneling current during needle formation. The number of extracted Si atoms was estimated from the STM images of the Si surface by measuring the area where Si atoms were removed during the voltage application. All the STM images were taken in constant current mode at a tip bias voltage of -2.0 V and a tunneling current of 0.2 to 0.5 nA.

### 3. RESULTS AND DISCUSSION

The voltage dependence of the sample nano-needle growth was measured by applying tip voltages between -3 and -10 V at a tunneling current of 0.5 nA. The results are shown in Fig. 1 for various duration times of applied voltage  $t_a$ . The most interesting feature is the threshold voltage of nano-needle growth between -6 V and -7 V. Although the needle did not grow below -6 V, growth suddenly began above -7 V and extended gradually with increasing voltage. Negative values appearing in the plots below -6 V indicate that holes were formed on the sample surface. Magnified STM images of the surface areas after applying voltages of -5, -6 and -10 V in Fig. 2. At an applied voltage of -5 V, a hole was observed which suggests that Si atoms were extracted from the surface. Si atom extraction by applying voltage pulses on the tip was reported,<sup>2)</sup> in which the threshold voltage for extraction was about 4 V regardless of the bias polarity. Since the extracted Si atoms were not redeposited around the hole, they might have been transferred either to the tip apex or to the vacuum. At applied voltages of -6 and -10 V, a nano-needle grew in the center of the hole. The voltage dependence of these surface modification results suggests that the surface Si atoms were extracted during the low-bias regime and were transferred to the tip, then were redeposited to the sample surface above the threshold voltage of approximately -6 V resulting in the needle formation.



Fig. 1. The voltage dependence of nano-needle growth height for various duration times of applied voltage.



Fig. 2. Magnified STM images of the sample surfaces where voltages were applied: (a) -5 V for 5s, (b) -6 V for 5 s, and (c) -10 V for 55 s.

The nano-needle formation mechanism will is analyzed based on this model, i.e. Si atom removal from the Si surface, transport to the tip apex, and redeposition to the sample surface. Assuming that the extraction of a Si atom takes place above particular threshold field  $E_t$ , the surface area where the electric field is higher than  $E_t$  under the tip is calculated by a simple model. The area S of the region, where the electric field generated by a point charge Q = CV at a distance d away from the surface is higher than  $E_t$ , is given by

$$S = \pi \left(\frac{Cd}{2\pi\varepsilon_0 E_t}\right)^{\frac{2}{3}} V^{\frac{2}{3}} - \pi d^2, \tag{1}$$

where  $\varepsilon_0$  is the dielectric constant of vacuum, and C is the capacitance between the tip and the sample. Equation (1) suggests that the needle height has a voltage dependence of  $V^{\frac{2}{3}}$ .

The area with extracted Si atoms was measured in STM images observed after applying various positive voltages. We used positive voltages because negative voltages produce needles on the sample surface, which makes it difficult to observe the surface surrounding the nano-needle due to the hindrance by the tip image. Kobayashi *et al.* reported that the conditions of Si atom extraction have almost no dependence on the voltage polarity,<sup>3)</sup> so reverse polarity should produce the same results. Si extracted areas were measured from STM images and the voltage dependences were derived, as shown in Fig. 3. Above the threshold voltage of about 5 V, the area increased with increasing voltage. The solid line in Fig. 3 is a fitting curve based on Eq. (1); it shows a good agreement with the experimental data.



Fig. 3. The voltage dependence of the Si extracted area. The solid line shows the fitting curve by the Eq. (1).



Fig. 4. The correlation between the atom extracted area and the nano-needle height.

The correlation between the area with extracted Si atoms and the nano-needle height is shown in Fig. 4, which shows that the area with extracted Si atoms has a linear dependence on the nano-needle height above 6 V. These results indicate that the needle height is almost proportional to the number of extracted atoms, because the area with extracted Si atoms is approximately proportional to the number of extracted Si atoms. Therefore, the material of the nano-needle can be originated from the Si atoms extracted from the surface.

The time dependence of nano-needle growth is shown in Fig. 5(a), where needles were formed by applying -7 and -10 V for 0 to 60 s. The growth of needles suddenly initiated in the first 1 s and terminated in about 20 s. The final height depended on the applied voltage. The time dependence of the extracted Si atom area was also measured using positive voltages as shown in Fig. 5(b). The number of extracted Si atoms had no dependence on the voltage application time. This result supports the Si atom extraction model, because once a needle has been formed, the tip is withdrawn from the surface to decrease the electric field at the region around the nano-needle below the threshold, so atoms can no longer be extracted.

Figure 6(a) shows the current dependence of the needle height measured for needles formed by applying tip voltages of -10 V at a tunneling current between 1 and 300 nA. The nano-needle height decreased as tunneling current increased and fell to almost 0 nm above a tunneling current of 200 nA, where the tip was often damaged and the apex shape completely changed. On the other hand, at a low current of less than 10 nA, the resulting nano-needle height dependence on current was almost negligible. The current dependence of the extracted Si atom area was measured for positive tip voltage, as shown in Fig. 6(b). The area slightly increased with increasing current, which can be explained as an enhancement of the electric field under the tip due to the tunneling gap narrowing with increasing current. The decrease in needle height with increasing current can be attributed either to the thermal diffusion of Si atoms due to Joule heating or to electromigration of Si atoms. The electromigration of Si atoms on Si(111) surface has been observed by STM, and Si atoms were found to migrate in the same direction as the current.<sup>4)</sup> Since the current flows from the sample to the tip in our work, electromigration mechanism is not valid. Therefore, the thermal diffusion model would be an appropriate explanation of the phenomena.

From these results, we propose Si atom extraction model for the needle formation process, which consists of the following three stages:

- (i) Extraction of Si atoms from the sample surface to the tip.
- (ii) Migration of Si atoms towards the highest protrusion of the tip apex.
- (iii) Redeposition of Si atoms from the highest protrusion of the tip apex to the sample surface.



Fig. 5. The time dependence of (a) the nanoneedle growth height and (b) the atom extracted area using positive tip voltages.



Fig. 6. The current dependence of (a) the nanoneedle growth height and (b) the atom extracted area using positive tip voltages.

Si atom migration in (ii) can be attributed to the surface diffusion due to an electric field gradient. Since there is a field gradient towards the tip apex on the tip surface, if Si atoms on the tip have dipole moments induced by the electric field, they should be attracted to the tip apex.<sup>5)</sup> Uchida et al. reported extraction and redeposition of Si atoms using an identical voltage pulse,<sup>2)</sup> which suggests that the Si transfer direction can be reversed due to the tip condition, as in (iii).

## 4. CONCLUSION

Nano-needle structures were formed on a Si(111) surface by applying negative ramp voltage of around -10V to a scanning tunneling microscope (STM) tip. Convolution image of needles on the sample surface and on the tip apex was obtained and the needle shape was estimated to be approximately 2 nm (5 nm at maximum) in diameter and around 10 nm in height. A most plausible model for the needle formation process consists of three stages: (i) Si atom extraction from the surface to the tip due to the high voltage application, (ii) migration of Si atoms to the tip apex, and (iii) redeposition of Si atoms from the tip apex to the sample surface, resulting in the formation of nano-needles.

### REFERENCES

- S. Heike, T. Hashizume and Y. Wada: Jpn.J.Appl.Phys., <u>34</u>, (1995) L1061.
- H. Uchida, D. Huang, J. Yoshinobu and M. Aono: Surf.Sci., 287/288, (1993) 1056.
- A. Kobayashi, F. Gray, R. S. Williams and M. Aono: Science, <u>259</u>, (1993) 1724.
- H. Tokumoto, K. Miki, and Y. Morita, Ultramicroscopy: <u>42-44</u>, (1992) 816.
- L. J. Whitman, J. A. Stroscio, R. A. Dragoset and R. J. Celotta: Science, <u>251</u>, (1991) 1206.