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Analysis of the process of anodization with AFM

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Abstract

Nanofabrication is necessary for building a nanodevice, and nanometer scale oxide structures are primary elements for achieving quantum effect. Local oxidation induced by electric field with AFM is a promising method. In this paper, an experimental setup with process monitor is established for analyzing the fabrication technology. Some oxide dots and lines on Si surface were fabricated using the conductive atomic force microscope, while the electric current between the tip and the sample is monitored. The process of anodization is analyzed based on the variational current. The electric charge dependence on the electric current plays an important part in the formation of oxide structures, thus the cross section of oxide dots or lines can be controlled by adjusting the current between the tip and the sample in the process of anodization.

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Keywords: AFM; Oxide structure; Nanofabrication; Electric current

1. Introduction

Nanofabrication is necessary for the study of nanodevice, and nanoscale oxidation structures are primary elements for achieving quantum effect. Scanning probe lithography could be the easiest method for achieving nanofabrication resolution, and has recently attracted much interest. Localized field-induced anodization on semiconductor and metal surfaces is a promising method for achieving nanoscale oxide. Since the first oxide induced by electric field with STM was put forward in 1990 [1], a few research groups have paid attention to the mechanism of the oxidation [2–6], the parameters of fabrication, and the application of oxide structures in some nanodevices such as the gate of an MOS transistor, point quantum devices or a single electron transistor (SET) [7,8]. Since the performance of conductive AFM is better than STMs on the stability and controllability of fabrication, most of researchers are interested in the study of anodization with AFM. Up to now,

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there have been some reports on AFM field-induced oxidation in contact, noncontact, and taping modes [9–11]. Different voltage waveforms, include constant, square wave, and sine wave, are applied to the fabrication [12–14].

However, the process of fabrication is rarely studied. In this paper, the electric current between the tip and sample was monitored the effect of different wave voltages. Therefore, the process of oxidation induced by electric field with AFM in air is analyzed, and a mechanism based on the electric current was described for better improvement of the control performance of this nanofabrication method.

2. Experiments

The experiments were performed with a Digital Instruments Multimode SPM. The fabricating voltage applied to the sample is from the output of an arbitrary wave generator Tektronic AWG2021, which is easy for adjusting the waveform, amplitude and frequency of voltages. Keithley 6517 A electrometer is inserted between the sample and AWG2021 for monitoring the electric current. The current data of 6517 A is input to a computer by GPIB interface. Fig. 1 shows the schematic diagram of the experimental system. The conductive probe is SC12/W_2C AFM probe from NT-MDT corp., and the radius of the tip is about 30 nm. In the process of fabrication, the AFM is set as the contact mode, and the feedback loop is kept at all times.

The experiment samples is n-type Si(1 0 0), which is passivated by 4% HF for removing the natural oxide on the surface.

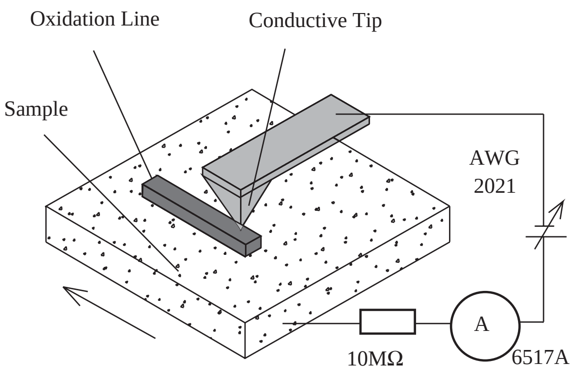

Fig. 1. Schematic diagram of the AFM field-induced fabrication system.

3. Results and discussion

Firstly, four dots are fabricated on the surface of n-Si(1 0 0) $5–8\times10^{-3}\,\Omega\,\mathrm{cm}$ by using different voltage waveforms with the maximum amplitude 7 V for analyzing the characteristics of electric current comparatively, shown in Fig. 2. The voltage waveforms, which include square, saw-tooth, triangle, and trapezoid, are described in Fig. 3, and are applied to the surface in clockwise. The electric current data monitored by 6517 A are described in Fig. 4.

Among the four modes, the peak of current induced by square waveform is minimum, and the current is faded down exponentially which demonstrates the mechanism of anodization. At other modes, the currents firstly have a quick increase, and then rise gradually. The reverse currents are

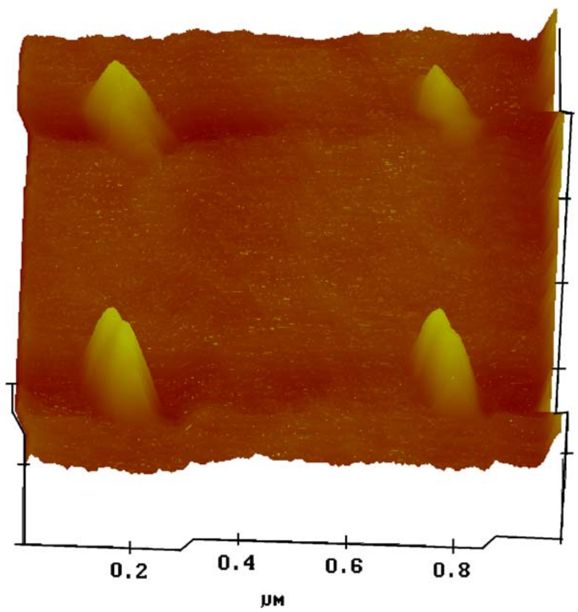

Fig. 2. Four dots fabricated with different voltage waveforms.

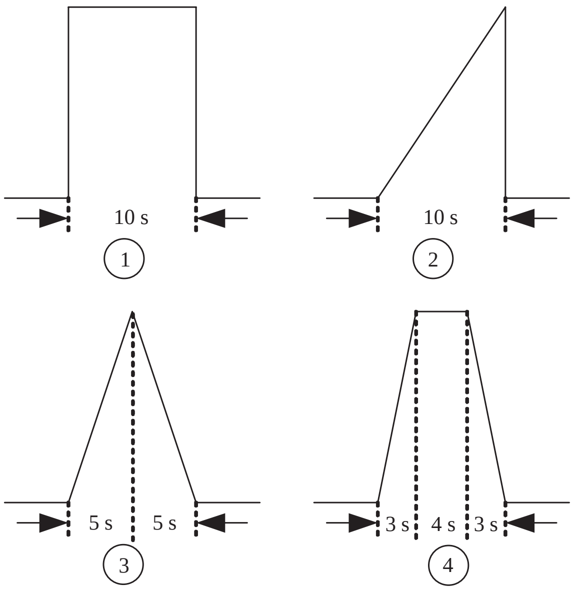

Fig. 3. Four voltage waveforms for oxidation.

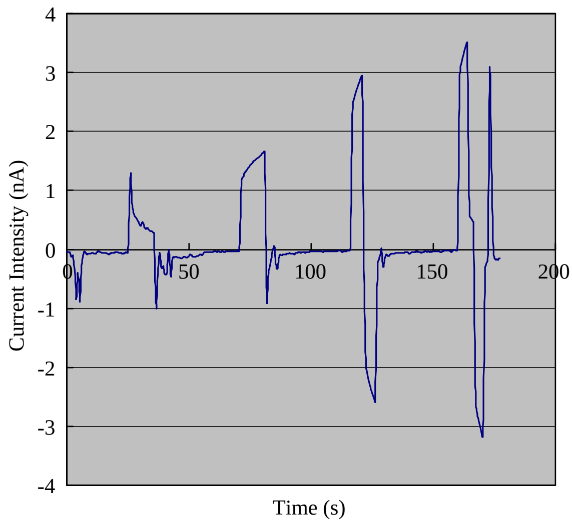

Fig. 4. Electric current monitored by 6715 A.

observed, when the voltages are removed (square and sawtooth wave) or decreased (triangle and trapezoid) gradually.

The above experimental phenomenon indicates that the electric charge plays an important part in the formation of oxide structures. The evidence is as follows:

1. The equation of the oxidation reaction on Si surface induced by electric field is

   $$Si + 2OH^- + 4H^+ \rightarrow SiO_2 + 2H^+$$

   OH^- is from the ionization of few nanometers water film on Si surface.
2. When a square voltage is applied, the maximum thickness of oxide will be reached in short time. The maximum thickness of oxide depends on the strength of electric field. When linear-gradual voltage is applied, the thickness of oxide is increased gradually with the increasing of voltage.
3. Since SiO_2 is an insulator, the tip, the insulator and the sample consists of a capacitor, which results in the charge and discharge of electron.
4. The capacitance is correlative to the thickness of oxide insulator. Since the thickness of oxide under the effect of square voltage is almost invariable after a moment, a typical charge curve of capacitor is observed. In the process of applying gradual voltage, the gradual-increase thickness of oxide results in the decrease of capacitance. At the same time, the voltage applied to Si surface is increased linearly. Thus, the currents have a slight raise under the compositive actions of the above two factors.
5. When the voltages are decreased gradually, the larger reverse currents are observed. When the voltages are removed, the amplitude of currents is lower than the formers. By comparing these current curves, we can know that the amplitude of currents depends on the descending slope of applied voltage. Thus, it demonstrates that the accumulation of electric charge exist on the process of electric-field-induced oxidation.
6. By comparing cross sections of four oxide dots, we can find that the volume of oxide dot by applying square wave voltage is larger than others three dots. The result validates that the anodization is only sensitive to the strength of electric field. In general, we can adjust the applied voltage, which is not equal to the electric field as a result of the changed capacitor and resistance.

7. The electric field is related to the charge and discharge of electrons, and these electric charges will diffuse to the adjacent area surrounded the center of the tip. Therefore, the electric field decreases gradually from the center of the tip. For this reason, the cross section of oxide dots or lines is approximately a triangle or a trapezoid.
8. We can control the charge and discharge of electrons by adjusting the voltage waveform with the aid of monitoring the current between the tip and the sample, thus the cross section of oxide structures can be forecasted and controlled in the process of anodization.

For understanding the effect of voltage amplitude, we adjust the maximum amplitude of applied voltages to 7.0, 6.0, 5.0, and 4.0 V sequentially. The voltage waveforms are same as the above experiment, and 16 dots are fabricated on Si surface. The electric current data monitored by 6517 A are described in Fig. 5. The current curves are very similar for each voltage waveform, and we find that the current increase along with the improvement of the amplitude of fabricating voltages for each voltage waveform. The thickness of these oxide dots rises with the increase of the amplitude of voltages. And the increase of maximum current and oxide thickness are linear with the increase of voltage amplitude.

Nanoscale oxide lines are primary elements for achieving quantum effect, and often are fabricated for making the insulating tunnel junction. The high aspect ratio is helpful for the study of nanodevice. In general, the fabrication process of oxide lines are as follows: (1) Moving the tip to the starting point of line, (2) applying the constant voltage to the sample or the tip, (3) moving the tip continuously along with the track of line, (4) removing the voltage at the end point of line. Although the amplitude of voltage, the moving speed of the tip, and the humidity can be adjusted for obtaining good results, adjacent point is gradually oxidized before moving the tip center to its top. For one point of the line, the fabrication voltage is equivalent to a gradual-increase voltage in the course of moving the tip.

By referencing the above analysis about the current, we understand that the square wave is better on reducing the diffusing of electric charge than other waveforms.

For solving this problem, we apply square wave voltages with the frequency of 200 Hz and the impulse ratio of 80% for fabricating lines. Fig. 6(a) is the cross section of oxide lines fabricated by constant voltages, which are 3.0, 4.0, 5.0, 6.0, and 7.0 V from left to right. Fig. 6(b) is cross section of oxide lines fabricated by square wave voltages, are 10.0 and 8.0 V from left to right, respectively, and the tip moves along these lines at speed of 0.5 μm/s. The aspect ratio of oxide lines are improved under the effect of square wave voltages by comparing Fig. 6(b) with Fig. 6(a). There exists a

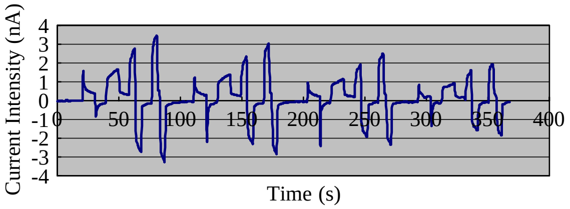

Fig. 5. Electric current by applying four voltage waveforms with the amplitude 7, 6, 5, and 4 V.

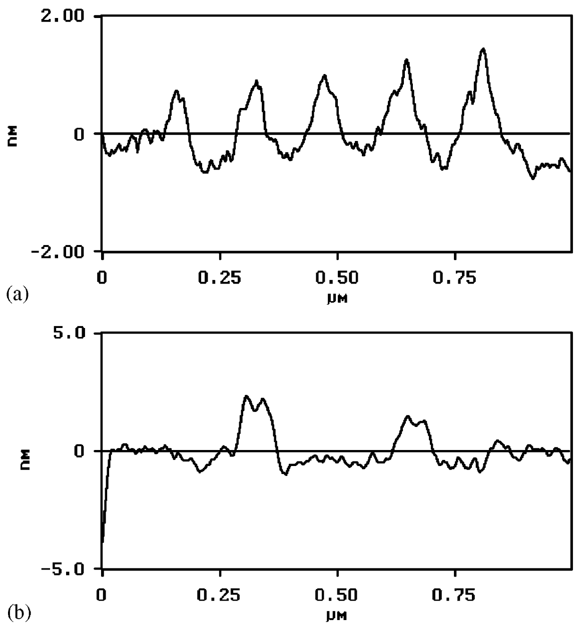

Fig. 6. Cross section of oxide lines fabricated by (a) constant voltages, (b) square wave voltages.

very fast accumulation and discharge of electric charge in the process of applying this square wave voltage, and the side diffuseness of electric charge is reduced. Then the transverse size of silicon oxide is reduced, and the aspect ratio of two edges is improved.

In addition, we find that the frequency of square wave is correlated with the moving speed of the tip, and the relationship will be described in the next paper.

4. Conclusions

The fabrication process of oxide structures on Si surfaces using a conductive AFM in air was investigated and analyzed based on monitoring the current between the tip and the sample in this paper. The current reflects the process of charge and discharge, which is related to the strength of electric field between the tip and the sample. It is useful to study on the mechanism and fabrication technology of electric-field-induced oxidation by monitoring the real-time electric current. The electric charge dependence on the electric current plays an important part in the formation of oxide structures, thus the cross section of oxide dots or lines can be controlled by adjusting the current between the tip and the sample in the process of anodization. The aspect ratio of oxide structures is improved by applying square wave voltage.

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