SPM local oxidation nanolithography with active control of cantilever dynamics

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Abstract. Local oxidation nanolithography using scanning probe microscope (SPM) has enabled us to fabricate nanometer-scale oxide wires on material surfaces. Here, we study tapping mode SPM local oxidation experiments for silicon by controlling the dynamic properties of the cantilever. Dependence of feature size of fabricated oxide wires on the amplitude of the cantilever was precisely investigated. The quality factor (Q) was fixed at a natural value of ~500. By enhancing the amplitude of the cantilever, both width and height of fabricated Si oxide wires were decreased. With the variation of the amplitude of the cantilever from 0.5 V to 3.0 V (DC voltage = 22.5 V, scanning speed = 20 nm/s), the feature size of Si oxide wires was well controlled, ranging from 40 nm to 18 nm in width and 2.3 nm to 0.6 nm in height. Standard deviation of width on Si oxide wires formed by tapping mode SPM is around 2.0 nm, which is smaller than that of contact mode Si oxide wires. Furthermore, the variation of the oscillation amplitude of the cantilever does not affect the size uniformity of the wires. These results imply that the SPM local oxidation nanolithography with active control of cantilever dynamics is a useful technique for producing higher controllability on the nanometer-scale fabrication of Si oxide wires.

1. Introduction

Local oxidation nanolithography using scanning probe microscope (SPM) has enabled us to fabricate nanometer-scale oxide wires on material surfaces [1]. It is well known that an external voltage applied between SPM tip and sample locally induces the anodic oxidation on the surface. Silicon [2], titanium [3] and niobium [4] surfaces have been locally oxidized by the SPMs such as scanning tunneling microscope (STM) and atomic force microscope (AFM). It is considered that SPM local oxidation is a novel nanolithography technique for fabricating single-electron transistors (SETs) [5], metal-insulator-metal (MIM) diodes [6] and planar-type ferromagnetic tunnel junctions [7].

Several SPM modes, such as contact, noncontact and tapping, have been used to perform local oxidation nanolithography. Local oxidation using contact mode is the most extended method for SPM nanolithography techniques. It has been reported that the use of tapping mode SPMs to locally oxidize the surfaces is also available as a nanolithography tool in order to obtain a precise control of the oxidation on the nanometer scale and to improve the reproducibility of the oxidation process [8-10].

On the size uniformity of Ni and Fe oxide wires, we have reported that the tapping mode SPM local oxidation produces higher controllability than that of contact mode SPM technique [11]. It is
considered that the interplay between water meniscus and space charge build-up in the oxide is important for the stable oxidation. Space charge effect reduces the growth rate and interrupts the oxide growth. Since the excitation of the SPM tip at its resonance frequency causes the modulation of the electric field in the meniscus, the space charge within the oxide may be easily neutralized and released by the tapping mode SPM local oxidation. In this paper, we consider that the precise control of dynamic properties of the cantilever is important for improving the size controllability and uniformity of the fabricated oxide wires. Here, we study the size controllability and uniformity on the nanometer-scale fabrication of Si oxide wires by varying the dynamic properties of the cantilever.

2. SPM local oxidation nanolithography

Nanometer-scale structures fabricated by using SPM local oxidation have been mainly controlled by applied bias voltage, scanning speed and ambient humidity. The reaction mechanism of the SPM local oxidation is considered to be the anodic oxidation. Since the local oxidation using SPM requires the field-induced formation of water meniscus between a conductive SPM tip and material surface, a negative bias voltage is applied to the SPM tip. Consequently, the water meniscus connects the SPM tip and the material surface and creates oxyanions (OH\(^-\), O\(^-\)) from water molecules. These oxyanions act as reaction species in order to form the oxide. In this way, one can easily obtain the oxide wires with a feature size of around 10 nm, which suggests the spatial dimension of water meniscus [12, 13].

In contact mode SPM local oxidation, it has been reported that growth rate of the fabricated oxide in case of applying AC bias voltage to the SPM tip was larger than that of DC bias voltage [14]. This suggests that space charge (H\(^+\)) build-up within the fabricated oxide during the SPM local oxidation is interrupted by the voltage modulation, resulting in the increase of the growth rate. On the other hand, SPM local oxidation nanolithography using tapping mode is a useful technique for precise control of the feature size of the oxide wires. Since the SPM tip is excited at its resonance frequency for the tapping mode experiments, it may be possible to neutralize and release the space charge within the oxide [11].

Figure 1 shows a schematic of local oxidation nanolithography process using tapping mode SPM. In tapping mode SPM local oxidation, the cantilever is driven at its characteristic resonance frequency and has a free air amplitude determined by drive amplitude, spring constant and quality factor (Q) of the cantilever’s mechanical resonances. The quality factor of the resonance is defined by \( Q = \frac{m}{\zeta} \), where \( \zeta \) is the damping factor of the cantilever. In this study, Q of the cantilever was naturally to be \( \sim 500 \) and the amplitude of the cantilever was varied ranging from 0.5 V to 5 V. This control is achieved via the implementation of a

![Figure 1. Schematic of local oxidation nanolithography using tapping mode SPM.](image-url)
positive feedback loop that enhances the effective quality factor and the amplitude of the cantilever. It has been reported that the method can improve the force sensitivity of dynamic force microscopy (DFM) including tapping mode in heavily damped environment [15]. Furthermore, quality factor enhancement in magnetic force microscope (MFM) allows the sensitive measurement of the magnetic domain structures in ambient [16]. Since the effects of the dynamic properties of the cantilever are still not clear on the tapping mode SPM local oxidation experiments, the dependence of the feature size of the fabricated oxide on the amplitude of the cantilever is investigated by varying the excitation of the cantilever.

3. Result and discussion
The experiments were performed with an SPM operated in tapping mode (SPA400/SPi4000, SII Nano Technology) in ambient air. The relative humidity was kept at ~30 %. The sample was p-type silicon substrates with a resistivity of ~1 k·cm. Doped n⁺-type silicon cantilever was used. The spring constant and the resonance frequency of the cantilever were typically 40 N/m and 300 kHz, respectively.

The amplitude of the cantilever was changed from 0.5 V to 5 V by tuning the excitation voltage of the cantilever. Size (height and width) dependence of Si oxide wires, formed by changing the amplitude of the cantilever in the tapping mode SPM local oxidation, is shown in figure 2 (a). The width presented here indicates the full width at half maximum (FWHM) of the Si oxide wire. All SPM images in this study were taken in size of 1 x 1 µm². In the experiments, the scanning speed was set at 15 nm/s and the applied DC voltage was 20 V. The quality factor of the cantilever was fixed at a natural value of 454. With enhancing the amplitude of the cantilever, the size of the fabricated oxide wires was decreased. Figure 2 (b) also shows the width and the height of the oxide wires in figure 2 (a). By changing the amplitude of the cantilever from 0.5 V to 5.0 V, the feature size of the fabricated oxide was well controlled, ranging from 55 nm to 25 nm in width and 2.2 nm to 0.5 nm in height.

In addition, matrix data of the local oxidation experiments were taken with different DC voltages and amplitudes. The scanning speed and the quality factor were set at 20 nm/s and a natural value of 505, respectively. The DC bias voltage and the amplitude were varied from 20 V to 27.5 V and from 0.5 V to 3 V, respectively. Figure 3 (a) shows the SPM images of the matrix data and the size of the oxide wires with different DC voltages is also shown in figure 3 (b). Feature size of the oxide wires is decreased with the increase of the amplitude of the cantilever. Furthermore, as widely known in SPM local oxidation experiments, the size is also well controlled by changing the applied DC voltage.

![Figure 2](image_url)

**Figure 2.** (a) SPM images of tapping mode SPM local oxidation experiments performed for different amplitudes of the cantilever. Quality factor was fixed at a natural value of 454. DC bias voltage applied to the tip and scanning speed of the tip were set at 20 V and 15 nm/s, respectively. (b) Size dependence of Si oxide wires on the amplitude of the cantilever.
Figure 3. (a) SPM images of tapping mode SPM local oxidation experiments performed for different amplitudes and DC bias voltages. Quality factor and scanning speed of the cantilever were set at 505 and 20 nm/s, respectively. (b) Size dependence of Si oxide wires on the amplitude of the cantilever. Different DC bias voltages were applied to the SPM tip.

Figure 4 shows SPM images and cross sections along the X axis in (a) contact mode and (b) tapping mode SPM local oxidation experiments. The nine cross sections were taken at 110 nm intervals along the Y axis. To evaluate the size uniformity of the Si oxide wires, the standard deviation of width was determined from the cross sections. Since the contact mode SPM local oxidation with applying the voltage above 20 V induces the excess oxidation, the oxide wires in figures 4 (a) and 4 (b) show larger width and higher standard deviation compared to the tapping mode.

Figure 4. SPM images and cross sections of Si oxide wires fabricated by (a) contact mode and (b) tapping mode SPM local oxidation experiments. DC bias voltage and scanning speed of the cantilever were set at 5 V and 10 nm/s, respectively. The nine cross sections were taken at 110 nm intervals along the Y axis.
(b) were formed with a voltage lower than 20 V for the direct comparison between contact and tapping mode SPM local oxidation experiments. The standard deviation of the width on the contact and tapping mode experiments was 9.2 nm and 2.5 nm, respectively. Thus, the size fluctuation of the Si oxide wires is further suppressed in the tapping mode SPM local oxidation.

We consider that the larger amplitude of the cantilever may reduce the spatial dimension of the water meniscus formed between the SPM tip and the sample surface, resulting in the smaller resolution of the fabricated oxide. Additionally, the amplitude enhancement causes to decrease the average intensity of electric field between the tip and the sample. This also contributes to the improved size controllability with smaller resolution of the fabricated oxide. In the tapping mode SPM local oxidation with the modulation of the amplitude of the cantilever, since the SPM tip is excited at its resonance frequency, the space charge accumulated in the oxide is easily neutralized and released by the modulation of the electric field, resulting in the stable oxidation. Moreover, standard deviation of the width on the Si oxide wires in Figure 3 (a) was typically determined to be around 2.0 nm, so that the variation of the amplitude of the cantilever during the tapping mode SPM local oxidation experiments does not affect the size uniformity of the fabricated oxide wires. These results imply that the SPM local oxidation nanolithography with active control of the cantilever dynamics is a useful technique for producing higher controllability and uniformity of Si oxide wires.

4. Conclusions

In conclusion, tapping mode SPM local oxidation nanolithography was performed with active control of cantilever dynamics for the first time. Feature size of Si oxide wires was well controlled by varying the oscillation amplitude of the cantilever. Furthermore, size uniformity of the Si oxide wires does not depend on the modulation amplitude of the cantilever. In conventional SPM local oxidation, the size of fabricated oxide structures was mainly controlled by three parameters such as applied bias voltage, scanning speed and ambient humidity. In this study, for the improvement of size controllability of the Si oxide wires, the effects of the oscillation amplitude of the cantilever were studied from the viewpoint of a control parameter for tapping mode SPM local oxidation experiments. These results suggest that the tapping mode local oxidation nanolithography with active control of the cantilever dynamics is a useful technique for producing higher controllability and uniformity on the nanometer-scale fabrication of Si oxide wires.

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