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Tapping mode SPM local oxidation nanolithography with sub-10 nm resolution

S Nishimura¹, T Ogino¹, Y Takemura² and J Shirakashi¹

¹ Department of Electrical and Electronic Engineering, Tokyo University of Agriculture and Technology, Koganei, Tokyo 184-8588, Japan

² Division of Electrical and Computer Engineering, Yokohama National University, Hodogaya, Kanagawa 240-8501, Japan

E-mail: shrakash@cc.tuat.ac.jp

Abstract. Tapping mode SPM local oxidation nanolithography with sub-10 nm resolution is investigated by optimizing the applied bias voltage (V), scanning speed (S) and the oscillation amplitude of the cantilever (A). We fabricated Si oxide wires with an average width of 9.8 nm (V = 17.5 V, S = 250 nm/s, A = 292 nm). In SPM local oxidation with tapping mode operation, it is possible to decrease the size of the water meniscus by enhancing the oscillation amplitude of cantilever. Hence, it seems that the water meniscus with sub-10 nm dimensions could be formed by precisely optimizing the oxidation conditions. Moreover, we quantitatively explain the size (width and height) of Si oxide wires with a model based on the oxidation ratio, which is defined as the oxidation time divided by the period of the cantilever oscillation. The model allows us to understand the mechanism of local oxidation in tapping mode operation with amplitude modulation. The results imply that the sub-10 nm resolution could be achieved using tapping mode SPM local oxidation technique with the optimization of the cantilever dynamics.

1. Introduction

Scanning probe microscopes (SPMs) have been extensively used to perform nanometer-scale modifications on material surfaces [1]. Silicon is obviously one of the most interesting surfaces to modify at the nanometer scale, and several approaches have been reported [2, 3]. It is well known that the feature size of Si oxide structures is controlled by some of the relevant factors such as applied bias voltage between the tip and sample, scanning speed of the tip [4] and relative humidity (RH) of the atmosphere [5]. In addition, a key feature of SPM local oxidation is the formation of a water meniscus, which mainly determines the size of the oxide. Hence, the meniscus control has a primary importance for the local oxidation process [6]. Several SPM modes, such as contact mode [1], tapping mode [7], and non-contact mode [8] have been used to perform the local oxidation nanolithography. We have reported that the size of Si oxide wires was well controlled by the modulation of the oscillation amplitude of the cantilever, which is caused by the stable control of both the lateral dimension of water meniscus and the average intensity of electric field strength in water meniscus [9]. Here, we study the tapping mode SPM local oxidation with sub-10 nm resolution by tuning the local-oxidation conditions such as applied bias voltage to the tip, scanning speed of the tip, and the oscillation amplitude of the cantilever.

2. Tapping mode SPM local oxidation nanolithography

In tapping mode operation, the cantilever vibrates at its resonance frequency. Upon approaching the sample, the tip briefly touches, or taps, the surface at the bottom of each swing, resulting in a decrease in oscillation amplitude. As shown figure 1 (a), the feedback loop keeps this decrease at a preset value and a topographic image of the sample surface can be obtained. In the local oxidation process, an initial value of the oscillation amplitude is set to 120 nm or 204 nm with spring constant of 42 N/m or 2 N/m, respectively. Figure 1 (b) shows the dependence of the oscillation amplitude of the cantilevers on the output voltage of photo detector. The oscillation amplitude is changed by tuning the excitation voltage of the cantilever.

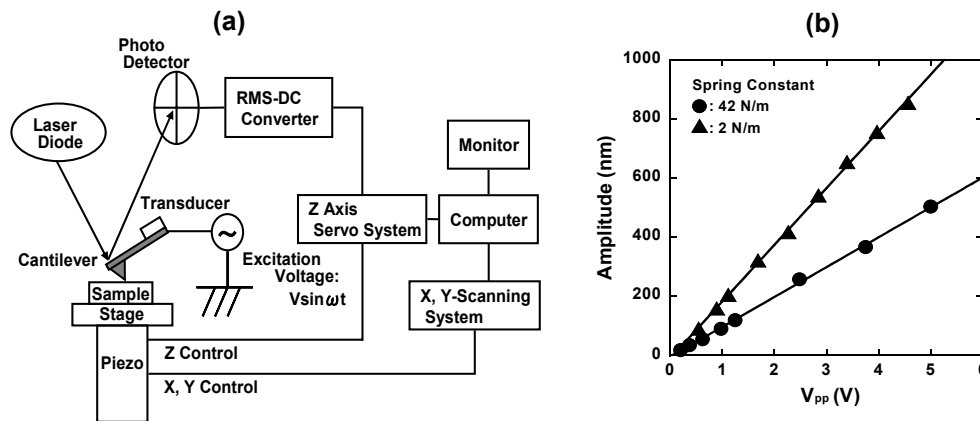


Figure 1. (a) Schematic diagram of a tapping mode operation system. (b) Oscillation amplitude of the cantilever as a function of output voltage (V_{pp}) of photo detector.

In contact mode SPM local oxidation, since a space charge (H^+) is built up within the fabricated oxides during the anodic oxidation, it is difficult to create and keep the stable local oxidation. On the other hand, it has been reported that SPM local oxidation nanolithography using tapping mode operation is a useful technique for a precise control of both size and uniformity of the Si oxide wires [9]. It may be possible to neutralize and release the space charges within the oxide because of the excitation of SPM tip at its resonance frequency. Furthermore, in tapping mode SPM local oxidation with the modulation of the oscillation amplitude of the cantilever, the size of the oxide wire is well controlled by varying the oscillation amplitude, suggesting that the lateral dimensions of the meniscus vary with the amplitude modulation [9]. In the present paper, by optimizing the control parameters such as applied bias voltage, scanning speed and the oscillation amplitude of the cantilever, we study the fabrication of Si oxide wires with sub-10 nm dimensions.

3. Results and discussion

The experiments were performed with an SPM operated in tapping mode (SPA400/SPI4000, SII NanoTechnology) under normal ambient RH conditions ranging between 20 % and 30 %. The sample was H-passivated p-type silicon with a resistivity of 1 $k\Omega \cdot cm$. Doped n^+ -type silicon cantilevers were used. The spring constant and resonance frequency of the cantilevers were typically 42 N/m and 300 kHz, respectively. The restoring cantilever force should be higher than the adhesion force due to the water meniscus [10]. For this reason, stiff and sharp cantilever was used in the experiments. The initial value of the oscillation amplitude of the cantilever was set at 120 nm. Figure 2 (a) shows an SPM image of a Si oxide wire formed by optimizing the oxidation conditions: the applied DC bias voltage of 17.5 V, the scanning speed of 250 nm/s and the oscillation amplitude of 292 nm. Figure 2 (b) shows the cross-sectional profiles of the Si oxide wire along the X axis. The ten point cross sections were taken at 50 nm intervals along the Y axis. The width presented here indicates the full width at half maximum (FWHM). The average width and standard deviation (STD) of the width were estimated by the cross sections, which were 9.8 nm and 1.9 nm, respectively. The size fluctuation of the Si oxide

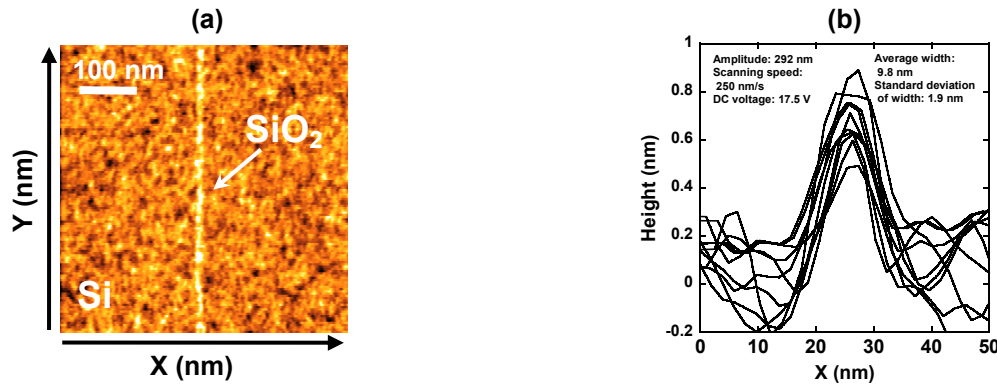


Figure 2. (a) SPM image and (b) cross section of Si oxide wire with sub-10 nm dimensions. Local oxidation was performed by optimizing the control parameters (DC bias voltage = 17.5 V, scanning speed = 250 nm/s, oscillation amplitude = 292 nm).

wire was further suppressed than that of contact mode experiments (STD of width = 9.2 nm) [9]. Furthermore, the minimum FWHM in the ten cross sections was 8.5 nm.

Figure 3 shows a simple model of tapping mode SPM local oxidation. The lateral dimensions of the water meniscus could be decreased by increasing the tip-surface separation. Amplitude modulation of the cantilever causes the cyclic meniscus formation between the tip and sample surface. Anodic oxidation occurs within the water meniscus, so that the oxidation time τ becomes discrete. In this situation, it seems that the average lateral dimension of the water meniscus during the oxidation was determined to be below 10 nm, resulting in the formation of Si oxide wire with sub-10 nm dimensions. Moreover, the amplitude enhancement over the maximum length of stretched water meniscus D causes to decrease the average intensity of electric field between the tip and sample. This also contributes to the improvement of size controllability of the oxide with smaller dimensions.

In addition, figures 4 (a) and 4 (b) show the size dependence of Si oxide wires on the amplitude of the cantilever under the applied voltage of 20 ~ 25 V and scanning speed of 20 nm/s. Open squares, circles, and triangles represent the experimental data. As mentioned above, the size is inversely proportional to the amplitude. Here, we propose an analytical model of tapping mode SPM local oxidation with respect to the oxidation ratio R . R is defined as $(\tau/T) \times 100$ (%), where τ is the oxidation time and T is the period of cantilever oscillation. Since τ clearly depends on the D , the oxidation ratio R is expressed as $((1/\pi) \times \sin^{-1}((2D-A)/A) + 0.5) \times 100$ (%), where A is the amplitude of the cantilever oscillation. The experimental data in the figures were fitted with the model based on the oxidation

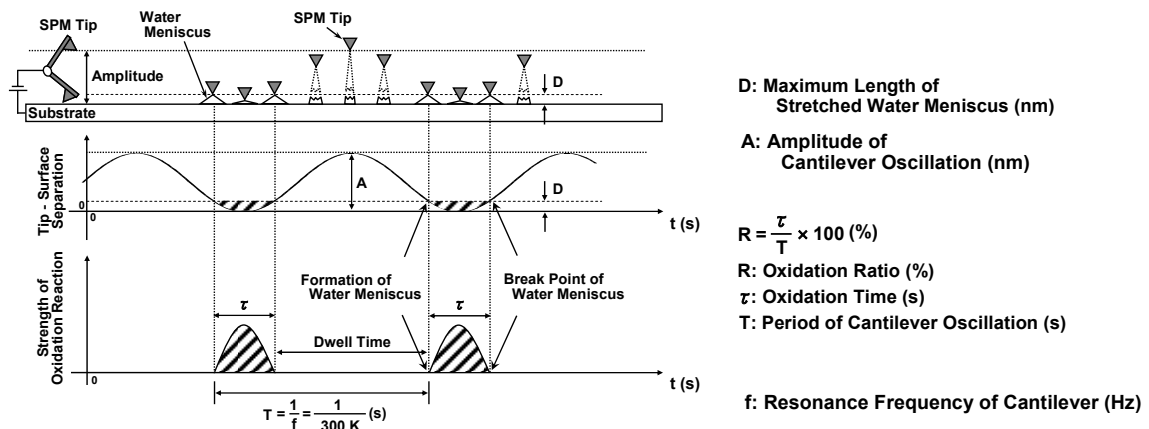


Figure 3. Model of tapping mode SPM local oxidation. The oscillation of the cantilever induces the cyclic formation of the water meniscus. The anodic oxidation occurs within the water meniscus and becomes discrete.

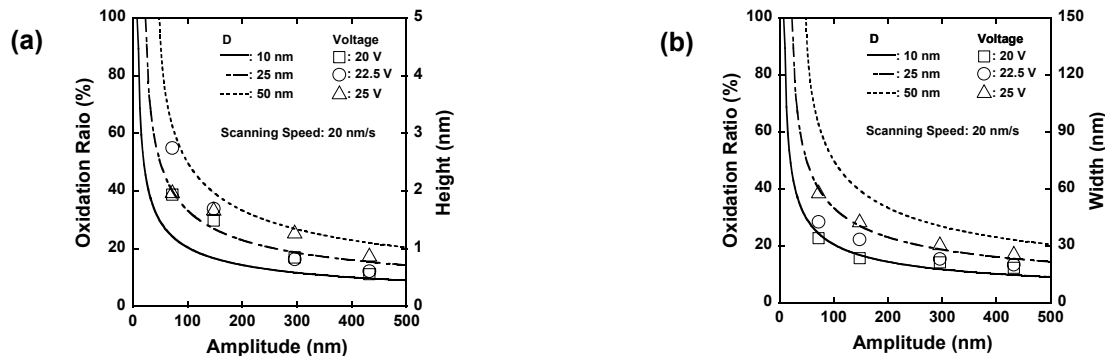


Figure 4. (a) Oxidation ratio and height as function of oscillation amplitude. (b) Oxidation ratio and width as function of oscillation amplitude. Oxidation ratio is calculated using the formula. Height and width are obtained from the oxides formed by tapping mode experiments.

ratio. When the D ranges from 10 nm to 50 nm, this model provides a good fit to the experimental data. In noncontact mode SPM local oxidations, the average electric field for the formation of the water meniscus is deduced as a function of the tip-surface distance and remains roughly constant ~ 1.3 V/nm [11]. Using the assumptions on the average electric field, the tip-surface distance of 26 ~ 32 nm is determined under the applied voltage of 20 ~ 25 V and is in good agreement with the D fitted in figures 4 (a) and (b). Furthermore, in our fitting procedure, 5 nm height and 150 nm width correspond to the oxidation ratio to be 100 % and are consistent with the size of the Si oxide formed by contact mode experiments [12]. This suggests that contact mode SPM local oxidation has no dwell time and shows the 100 % oxidation ratio.

4. Conclusion

Nanometer-scale modification of Si surfaces has been demonstrated using the tapping mode SPM local oxidation with the amplitude modulation. By optimizing the control parameters such as applied bias voltage to the tip, scanning speed of the tip and oscillation amplitude of the cantilever, the fabrication of Si oxide wires with sub-10 nm dimensions was achieved. The average width and minimum FWHM of the Si oxide wire were 9.8 nm and 8.5 nm, respectively. Furthermore, we proposed a simple model of tapping mode SPM local oxidation based on the oxidation ratio. Relations between the oxidation ratio and the oscillation amplitude were investigated and were consistent with the experimental results. These results imply that the tapping mode SPM local oxidation is suitable for producing the well-controlled Si oxide with sub-10 nm dimensions.

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