Velocity controlled anodization nanolithography with an atomic force microscope using Faradaic current feedback

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(Received 9 January 2007; accepted 29 January 2007; published online 7 March 2007)

A technique, called velocity controlled anodization nanolithography, is presented that ensures line continuity during atomic force microscope based local anodic oxidation on silicon. Spontaneous current spikes disrupt the generation of uniform silicon oxide patterns during lithography at low humidity. Varying the translational speed during lithography in response to the current fluctuations enables the formation of a more complete and continuous oxide layer. The velocity corrections as a result of control are able to maintain constant current flow through the tip-sample interface. The authors demonstrate that this method is effective for in situ quality control. © 2007 American Institute of Physics. [DOI: 10.1063/1.2711377]

The atomic force microscope (AFM) has become an indispensable tool for nanoscale manipulation and analysis. A multitude of experimental conditions affect the resulting oxide formation in both contact and noncontact modes of AFM operation, most important of which are voltage bias, humidity, and tip dwell time or write speed. In order to better understand the process by which the oxide forms, researchers began to monitor the current during lithography and detected current flow on the order of picoamperes. More recently it was determined that it is Faradaic in nature, which has led to an even better understanding of the physics behind the anodization process. In numerous instances, local anodic oxidation has been used to construct nanoscale electronic devices and as a mask for further processing. Critical to the creation of these devices and masks is the generation of a uniform and consistent oxide layer. Feedback of current through the tip during anodization using voltage modulation has been studied to increase line uniformity, which delivers a constant current to the system and a varying field. We outline here an alternative method in which current flow is controlled by velocity modulation. This allows for uniform voltage and current during the process so the power during lithography stays constant. In addition, the controller provides for real-time quality control during lithography that maintains uniform oxide formation. We believe this to be a unique demonstration of AFM based nanolithography that can detect and counteract potential defects in pattern formation before they are created.

Experiments were conducted on a custom nanolithographic platform. The substrates used in the experiments were cleaved from a 3 in. n-type phosphorous doped Si(110) wafer with a resistivity of 5–10 Ω cm (Montco Silicon Technologies). The samples were cleaned in a solution of 70% H2SO4 and 30% H2O2 at 80 °C for 15 min to remove contamination. Triangular Si3N4 cantilevers that have a nominal force constant of 0.58 N/m were used for the experiments (Veeco Metrology). These tips were coated with 30 nm of evaporated Ti at 2 Å/s. Current amplification is accomplished through the use of an instrument amplifier (6485 Picoammeter, Keithley Instruments).

An initial experiment involved the writing of a series of lines at various speeds during which the current was recorded (Fig. 1). It has been demonstrated that AFM based anodic oxidation at lower relative humidity (15%–45%) is able to produce thinner oxide lines. This fact is important to maximize the spatial resolution of AFM based anodic oxidation. When creating oxide lines at a constant velocity and voltage at low humidity levels, there are periods of discontinuity in the resulting oxide patterns, as experienced in our work and reported elsewhere. Figure 2 shows an example of what is observed; the figure depicts qualitative incompleteness within the series of lines. These features have an average height of 4.2±1.1 nm and width of 167±45.0 nm. Most notable is the rms variance in the height and width for the incomplete lines at y=2 and 4 µm, which is 1.4 and 1.7 nm in height and 58.8 and 70.2 nm in width, respectively. In contrast, the more uniform lines at y=1 and 3 µm have variances of 1.0 and 0.5 µm in height and 23.6 and 27.4 nm in width, respectively. Figure 3 is a plot of both the current and height trace recorded during lithography and imaging of the line at y=4 µm in Fig. 2. There is a distinct and coincident point along the length of the line in which the height and current undergo a sudden change. The change in average height of 1.8±0.8–4.9±1.0 nm happens at the pre-
cise moment when the current falls down to a measurable level of \(3.3\pm1.3\) pA at an \(x\) position of 2.3 \(\mu\)m. Before this point the amplifier is saturated due to currents greater than the dynamic range \(>2.1\) nA. Similar current and height characteristics are present in the line at \(y=2\) \(\mu\)m. The oxide height is increased almost threefold when the current drops down to the picoampere range. Conversely, the lines at positions \(y=1\) and 3 \(\mu\)m have uniform current signatures in the low picoampere range (average of \(5.6\pm3.1\) pA) and exhibit more uniform oxide.

The linear relationship between current and speed (Fig. 1) demonstrates that velocity modulation can be used to control the current through the tip-sample junction. A feedback controller was designed that compares measured current with a reference value and the error signal is used to command tip position based on the data. A similar set of four oxide lines was created using the velocity controller with a set point of 6.0 pA within 3 min of the original experiment.

![FIG. 1.](image1.png)

**FIG. 1.** (Color online) Plot of current through the tip as a function of velocity during line fabrication. The experiment was run at 9 V bias, 5 nN, and 20% RH. The current and height are averaged over the length of the oxide lines. The rms deviations for the data are represented by the bounds shown. The legend displays the equations of linear fits to the data as a function of the tip velocity.

A qualitative look at the deposited lines in Fig. 4 shows that they are more uniform than those formed in Fig. 2. These features have an average height of \(4.9\pm0.6\) nm and width of \(174\pm20.7\) nm. The inconsistencies in Fig. 2 represent a significant portion of the total oxide deposited. The average height and width variances over all deposited oxide in the constant velocity case are 1.1 and 45.0 nm, respectively. In contrast, the velocity controlled case has an average variance in height of 0.6 nm and an average variance in width of 20.7 nm over the data. The variances in the constant velocity case amount to 26.1% of the average height and 27.0% of the average width of the deposited features. Using velocity control, the variance is reduced to 12.2% of the average height and 11.9% of the average width. This demonstrates a significant improvement (more than a factor of 2) in both line height and width uniformity.

![FIG. 2.](image2.png)

**FIG. 2.** Contact AFM image of a series of four lines written at 9 V bias, 0.32 \(\mu\)m/s, 5 nN, and 20% RH. As can be seen in the image, the first and third lines (at \(y=1\) and 3 \(\mu\)m, respectively) appear qualitatively complete. The second and the fourth lines (at \(y=2\) and 4 \(\mu\)m, respectively) have noticeable discontinuity. Lighter shade corresponds to taller height.

![FIG. 3.](image3.png)

**FIG. 3.** (Color online) Line height and recorded current associated with the line at \(y=4\) \(\mu\)m in Fig. 2. The height in the early part of the line averages 1.8\(\pm0.80\) nm (current unrecordable) and in the later part, starting at \(x=2.3\) \(\mu\)m, is 4.9\(\pm0.98\) nm (current is 3.3\(\pm1.3\) pA).

![FIG. 4.](image4.png)

**FIG. 4.** Contact AFM image of a series of four lines written with the velocity controller implemented at 9 V bias, 6.0 pA set point, 5 nN, and 20% RH. In contrast to Fig. 2, uniformity exists across the entire length of all lines. Lighter shade corresponds to taller height.
The spike in current indicates a drastic decrease in the size of the water meniscus at a lower relative humidity.\textsuperscript{16,20} We hypothesize that in the constant velocity case (Fig. 2) the meniscus breaks down unpredictably. This breakdown is associated with a corresponding increase in current and a decrease in oxide height and width. The spike in current indicates a drastic decrease in the size of the water meniscus. Low humidity levels force the water adsorbed on the surface to be more confined in isolated regions on the substrate. With the meniscus gone or severely depleted in volume, no oxide can form due to the lack of oxygen ions. When this happens, it is hypothesized that the current flowing through the tip switches from Faradaic in nature to electronic (Ohmic and tunneling), resulting in larger current values.\textsuperscript{13} The lack of oxide growth results in less Ohmic resistance between the tip and the sample; the observed current increase is sustained as a result. An indeterminate point is reached in which the water returns to the tip-sample interface. When water is reencountered (x = 2.4 μm in Fig. 3) the meniscus reforms as demonstrated by a return to the low current regime. This indicates the return of Faradaic current flow and the formation of a larger volume of oxide. Looking at the effects of the implemented controller in Fig. 5, when the current begins to rise a change in tip direction and the corresponding change in velocity are able to reestablish Faradaic current flow. This serves to maintain a stable water meniscus and ensure uniform oxide formation with significantly less variance in the height and width of resulting features.

We have developed a feedback control methodology that not only is able to provide for quality control during lithography but also ensures constant power supply to the anodization reaction. Through Faradaic current monitoring during lithography, detrimental current fluctuations are counteracted by changes in the tip velocity. This process ensures that a continuous oxide pattern is formed. We have demonstrated that the average height and width variances as a percentage of the resulting feature dimensions were improved by a factor of 2 between two experimental anodization lithography runs that had identical average speeds, humidity levels, and bias voltages that were executed within 3 min of one another. We believe that these results demonstrate the validity of this method which provides for quality control during AFM based local anodic oxidation.

This work was supported by the NSF under NIRT DMI-0609265.