Step-In Mode NC-AFM Using a Quadrature Frequency Demodulator for Observing High-Aspect Ratio Structures in Air*

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We have investigated step-in mode non-contact atomic force microscopy (NC-AFM) for precise measurement of fine and steep structures having high aspect ratios. We have proposed piconewton controlled step-in mode AFM using NC-AFM to suppress bending and slipping of the probe on a slope. We have constructed a prototype of the step-in mode NC-AFM using a quadrature frequency demodulator for detecting the resonant frequency shift of the cantilever. Experiments revealed that the system was able to perform step-in mode NC-AFM even in air. We obtained a faithful AFM image of the steep structure of a dry-etched Si pattern without bending or slipping of the probe at approximately 2-3 pN using a sharp, slim probe, as compared with a step-in mode contact AFM image obtained at 1, 5, and 10 nN. [DOI: 10.1380/ejssnt.2011.122]

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I. INTRODUCTION

The capacity of Si memory devices is increasing with the packing density of memory cells, which is being advanced by the expansion and development of information technology. This progress has enabled the device structure to become small and have a high aspect ratio. The roadmap requires the fabrication of very fine hole patterns with a diameter of less than 60 nm, a depth of greater than 700 nm, and a structural aspect ratio of greater than 10. In addition, evaluation of the roughness and cross-sectional profile of fine patterns is required for advanced fabrication processes. At present, critical dimension scanning electron microscopy (CD-SEM) is used to measure some dimensions of large-scale integration (LSI) patterns. However, CD-SEM is insufficient for evaluating the roughness and cross-sectional profile. This is because SEM contrast is not proportional to the slope angle of the sample. In order to perform faithful three-dimensional (3D) measurements, nanometer-resolution atomic force microscopy (AFM) [1] is required.

On the other hand, in the measurement of steep structures, a sharp, slim probe is required, such as a carbon nanotube. However, such probes encounter problems such as bending and slipping, which introduce serious errors. In order to achieve faithful measurement using the probe, we have developed a step-in mode technique [2, 3]. The present research has demonstrated that the proposed method is suitable for CD-AFM and prevents bending of the probe by maintaining a constant gap between the probe and the sample surface during probe scanning in conventional AFM methods such as contact mode [1], non-contact mode [1, 4, 5] and tapping mode AFM [6]. In addition, we previously proposed inclination AFM for faithfully measuring steep structures [9]. However, a number of technical issues remain when using a sharp, slim probe for faithful measurement of very steep structure patterns with a high aspect ratio, even in the step-in mode. We have reported probe bending and slipping on steep structures which caused the position error in the AFM measurement [2, 8].

In the present paper, we describe the force dependence on position error due to bending and slipping of the probe, piconewton control in the attractive force region using the NC-AFM technique with a quadrature frequency demodulator, and faithful measurement of a steep dry-etched Si structure using a sharp, slim probe.

II. FORCE DEPENDENCE ON POSITION ERROR IN AFM

In a previous paper [8], we presented an analysis of probe bending and slipping on an inclined slope. Using a simple model such as a step-in mode AFM technique to prevent deflection of the probe and generation of friction due to scanning, we were able to calculate the position error under a constant force $F_c$ while controlling the probe by means of force balances.

When the probe slips and is twisted on a slope, the errors $\Delta r$ (parallel component) and $\Delta z$ (perpendicular component) are given by the following equations:

$$\Delta r = \frac{F_c}{k_t} \sin \theta - \eta \cos \theta$$

$$\Delta z = \frac{F_c \cdot \tan \theta}{k_z}$$

where $E$ is Young’s modulus, $\eta$ is the friction coefficient, $\theta$ is the slope angle, $k_t$ is the torsion spring constant of the probe, $\ell$ is the length of the probe, and $\gamma_o$ is the probe aspect ratio. The errors $\Delta z$ are calculated as a function of the constant force controlled in the AFM, as shown in Fig. 1. When the slope angle $\theta$ is 85 degrees, and $\gamma_o$ is 5, $\Delta r$ and $\Delta z$ are approximately 0.01 nm and 0.8 nm, respectively, at 10 nN. Furthermore, when $\theta$ is 88 degrees, $\Delta r$ and $\Delta z$ are approximately 0.08 nm and 2 nm, respectively, at 10 nN. When $\theta$ is 88 degrees and $\gamma_o$ is 10, $\Delta r$ and $\Delta z$ are approximately 0.8 nm and 20 nm, respectively, at the force (10 nN). Suppressing the position error to less than 1 nm requires a constant force of less than 100 pN. In practice, piconewton control cannot be achieved in the repulsive force region. The reason for this is that the force is too weak to control it because

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the detection of small deflections of a practical cantilever (small spring constant of 0.1 N/m) of within 0.1 nm is difficult for 100 pN. Therefore, the control must be performed in the attractive force region. As described above, in the present study, we must control the constant force to be as small as possible in the attractive force region for CD-AFM, when using a sharp, slim probe with $\gamma_a = 5$ to 10. Furthermore, we must suppress the force to be less than 10 pN for the case in which $\gamma_a = 20$.

III. PROTOTYPE STEP-IN MODE NC-AFM

We have constructed a prototype step-in mode NC-AFM using a quadrature frequency demodulator instead of a phase locked loop (PLL) demodulator [5] as a resonant frequency detector. The quadrature frequency demodulator prevents the AFM system from being stopped as a result of non-recovery of the frequency-voltage conversion after the probe contact and release of the sample in the PLL demodulator. We improved the step-in mode contact AFM system to operate in non-contact mode. A block diagram of the prototype step-in mode NC-AFM system is shown in Fig. 2. A resonant vibration was applied to the probe and a quadrature frequency demodulator was added to the AFM system.

The quadrature demodulator consists of a phase converter of the input signal, an analogue multiplier of the input and converted signals, a low pass filter, and an amplifier, as shown in Fig. 3. We continued detect changes in the resonant frequency of the probe without stopping the conversion. Figure 4 shows one of frequency-voltage (FV) conversion properties of the demodulator. The curve is

FIG. 1: Position errors $\Delta r$ and $\Delta z$ of the probe caused by nano-bending and slipping of high-aspect-ratio probes on a slope. (a) Illustration of nano-bending and slipping. Results obtained at slope angles of (b) 88 degrees and (c) 85 degrees.

FIG. 2: Schematic diagram of the prototype step-in mode NC-AFM system.

FIG. 3: Schematic of quadrature frequency demodulator.

FIG. 4: Typical frequency-voltage conversion properties of quadrature frequency demodulator.
FIG. 5: SEM image of sharp, slim probe, which was fabricated by ion milling.

FIG. 6: Typical SEM images of sample coated with thin Au film. (a) Top view. (b) Cross-sectional view.

seen to have an S-shape. This property can continue to detect the frequency even at such a contact. The figure shows typical property. The resonant base frequency is approximately 139.8 kHz, the conversion band width is approximately 1.3 kHz, and the FV conversion is approximately 26 mV/Hz.

In step-in mode operation, (1) the probe is made to approach the sample surface and to control the force gradient constant after xy-scanning has completely stopped, (2) the probe is moved stepwise from pixel to pixel just after gap control is stopped, and it is lifted up from the sample surface, and (3) a step-in mode NC-AFM image is produced by repeating steps (1) and (2) while xy-scanning the probe over the surface [2, 3]. This sequence is performed by modifying the software in the AFM system. A sharp, slim Si probe with a spring constant of approximately 48 N/m was produced by ion-milling probe. The aspect ratio of the probe was in the range of 5 to 8. An SEM image of the probe used is shown in Fig. 5.

IV. EXPERIMENTAL RESULTS

We observed dry-etched silicon samples under a controlled force of from 1 to 10 nN by step-in mode contact AFM and under a controlled force of 2 pN by step-in mode NC-AFM. The recession distance from the sample surface was approximately 500 to 1,000 nm, depending on the depth of the grooves and holes and the thickness of the contamination layer. The typical time required to obtain a single 256 × 256 pixel image was approximately 10 min. We first measured dry-etched Si line and space patterns with various widths and depths. The SEM images in Fig. 6 show a typical structure having line and space widths of approximately 1 and 1.2 µm, respectively, and a depth of approximately 1 µm. The SEM image in Fig. 6(b) shows a cross-section of the structure. The intensities at the line edges (shoulder edges) are asymmetric because of the influence of the secondary electron detector position in SEM. The angles of the sidewalls have been confirmed by SEM to be symmetric, and are from 85 to 87 degrees in Fig. 6.

Step-in mode contact AFM measurements yielded images of the Si structure at constant forces of 1, 5, and 10 nN, as shown in Fig. 7. The figure shows that measurement errors occur at the shoulder edges due to probe bending and slipping. As the controlled force increased from 1 nN to 10 nN, the error became large, and the AFM images deteriorated to lose the shoulder edges largely. This sample had line and space widths of approximately 250 nm.

In the step-in mode NC-AFM measurements, we used an ion-milled Si probe with a probe aspect ratio of from 5 to 8, a spring constant of 48 N/m, and a resonant frequency of 221.5 kHz (Fig. 5). The system was operated under a set frequency shift of −66 Hz, and the oscillation amplitude of the probe was approximately 2 nm. Based on the frequency shift, we can estimate the force to be approximately 2 pN, assuming that the atomic potential is of Lennard-Jones type. We obtained the step-in mode NC-AFM image of the Si dry-etched structure shown in Fig. 8. The image and cross section show clear shoulder edges, as compared with the step-in mode contact AFM images.
AFM image obtained at 1 nN. This indicates that the piconewton control is effective for faithful measurement of high-aspect-ratio structures. When we measure such a high-aspect-ratio structure to within an error of 1 nm for CD-AFM, we must use the step-in mode NC-AFM and the sharp, slim probe with a controlled force of less than 100 pN, as shown in Figs. 1 and 8.

V. SUMMARY

We have experimentally investigated the force dependence on the measurement error in AFM due to nano-bending and slipping of a sharp, slim probe in step-in mode contact and non-contact AFM. The following results were obtained:

1. The position errors $\Delta r$ and $\Delta z$ increase rapidly with the controlled force. In order to carry out faithful measurements, we must control the force to within 100 pN using a sharp, slim probe while suppressing $\Delta r$ and $\Delta z$ to within 1 nm at a slope angle of 88 degrees.

2. We constructed a prototype step-in mode atomic force microscope capable of both contact and non-contact mode AFM in air, by the use of quadrature frequency demodulation.

3. We obtained step-in mode contact AFM images of dry-etched Si line and space patterns at 1, 5, and 10 nN and NC-AFM images at 2 pN.

4. In the step-in mode contact AFM measurements, deformation of the shoulder edge occurred at 1 nN. This method is thus not suitable for faithful AFM measurements.

5. In the step-in mode NC-AFM measurements, no deformation of the shoulder edge occurred at 2-3 pN. This method is therefore suitable for faithful AFM measurements.