Measurement of Frictional Forces using Atomic Force Microscopy

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Abstract

A new method is proposed for the calibration of frictional forces in atomic force microscopy. An angle conversion factor is defined using the relationship between torsional angle and frictional signal. Once the factor is obtained from a cantilever, it can be applied to others without additional experiments. Moment balance equations on the flat surface and the top edge of a commercial step grating are used to obtain the angle conversion factor. The proposed method is verified through another step grating test and frictional behaviors of Mica.

I. INTRODUCTION

Since it was invented, atomic force microscopy (AFM)[1-4] has been given a great deal of attention by researchers who study surface topography, frictional properties, magnetic properties and so on at the nanometer scale. At the beginning, most studies with AFM were to analyze surface properties and compare these relatively. Nowadays, many mechanical tests[5-10] are being performed by AFM and these results are quantitatively compared with experimental data obtained at the macro scale. However, because the data measured in AFM are the electronic signals for the forces and the surface shapes, the calibration of the signals is essential for quantitative analyses[11-18].

AFM determines the surface topography and the adhesive force (pull-off force)[19] by measuring the displacement[4,20] at the tip of a microfabricated cantilever spring. Knowledge of the spring constants (bending stiffness, k_b) of an AFM cantilever and of the vertical sensitivity of the PZT scanner allows the quantitative measurement of adhesive force and the quantitative analysis of mechanical tests in the vertical direction of a cantilever. Frictional signals in AFM are measured by the torsional displacement of cantilever (i.e. the displacement of laser spot on the detector by the twist of a cantilever). The torsional stiffness of cantilever can be calculated by classical mechanics[21,22] or simulations, but other information is not provided for the quantitative analysis in the lateral direction. Therefore, quantitative analysis using AFM for mechanical tests in the lateral direction such as frictional properties has not been practiced.

Some researchers have conducted the calibration for the quantitative measurement of frictional signals (voltage signal, mV) in AFM. These methods are somewhat complicated and any methods do not consider the dimensional properties of a cantilever which can significantly influence the results of experiments. In addition, whenever the dimensional and physical properties of the cantilever used in AFM are changed for other purposes, the same experiments should be repeated for the calibration in the lateral direction. Therefore, in this study, a calibration method using the angle conversion factor is suggested. Because the factor is a constant value in an AFM, the same experiments need not be repeated for different cantilevers. To calculate the angle conversion factor, the moment balance equations on step grating are used and frictional tests in AFM are performed. The proposed method is verified through another step grating test and frictional behaviors of Mica.

II. ANGLE CONVERSION FACTOR

In AFM, cantilevers with different dimensions and physical properties can be used for various purposes. Fig. 1(a) indicates tips of two cantilevers with different tip heights. Though the cantilevers are twisted by the same frictional force, the short tip has a smaller torsional angle than that of the long tip because of the different moment at O point. Fig. 1(b) shows the laser spots induced from each cantilever on the detector. The positions of the laser spots are differently located. Because displacements of the laser spot on the detector by the torsional angle of a cantilever make frictional signals, the signals for two different cantilevers are different. Thus, the calibration factor (called the "force conversion factor") of the frictional signal in AFM must be calculated separately for each cantilever because frictional signals produced by the same frictional force are different for each cantilever. Previous calibration methods were to obtain the calibration factor of the cantilever used in AFM experimentally. Thus, the same experiments should be repeated to obtain the calibration factor whenever the properties of the cantilever are changed.

In this study, the angle conversion factor is used for the calibration of frictional signals in AFM. Eq. (1) indicates the angle conversion factor which is the ratio of the torsional angle to the frictional signal. The angle conversion factor is constant for an AFM because the factor indicates the characteristic property of the detector in an AFM. When frictional tests are performed in AFM, the frictional force makes the torsional angle of a cantilever. This angle causes the displacement from the reference point on the detector (see Fig. 1(b)), which is expressed through the frictional signal (in unit, mV). If the displacement on the detector is same though the reference points are different, the frictional signal is always same for the AFM. Thus, the ratio (i.e. the angle conversion factor) of the displacement to the frictional signal is always constant for an AFM.

Eq. (2) indicates the relationship between the angle conversion factor and the force conversion factor. In Eq. (2), k_t is the torsional stiffness of the cantilever and H is the distance from the tip end to the center of moment. Because the angle conversion factor is constant for an AFM, once the angle conversion factor is obtained, the force conversion factors for any cantilevers can be calculated without repeated experiments by Eq. (2).

$$\eta = \frac{\theta_0}{V_0} \text{ [rad/V]} \tag{1}$$

$$\kappa = \eta \frac{k_t}{H} \text{ [}nN\text{/V]} \tag{2}$$

III. FRICTIONAL FORCE STEP EDGE CALIBRATION

A. Phenomenon at step edge and assumptions

Fig. 2 indicates a schematic diagram and an AFM image for a commercial step grating. In the schematic diagram, the step is vertical, but in an AFM image, it is not. This is caused by interaction between the tip radius and the step edge of grating. When a frictional test is performed on a step grating, the frictional signal and surface topography are shown in Fig. 3(a). The frictional signal on the flat surface of the step grating occurs from the twist of the cantilever by the frictional force and the normal force between the surface and the tip (stages I and III in Fig. 3(a)). The frictional signal (it is referred to the maximum signal) on the top edge of the step grating occurs from the twist of the cantilever by the frictional force and the normal force between the top edge of step and the tip (stage II in Fig. 3(a)). However, it is found that the maximum signal does not happen at the end of a tip because of the difference (about 40~70 nm) between the positions of maximum signal and of maximum height in Fig. 3(a). Fig. 4 is an SEM image of tip. Because the pyramidal tip is sharpened toward the tip apex, the side line is not straight. Thus, the difference in positions of maxima is considered to originate from the effect of the tip radius and the shape of the tip apex.

In the proposed calibration method, the angle conversion factor is calculated by the moment balance equations at O point of the tip in Fig. 5. For this calculation, the next three conditions are assumed;

1) Maximum signal happens at the end of a tip.
2) The side line of a tip is straight.
3) The relationship between the frictional signal (V) and the torsional angle (θ) of a cantilever is linear such as Eq. (3).

$$V_0 : V_1 = \theta_0 : \theta_1 \tag{3}$$

B. Moment balance equation on the step grating

Fig. 5 shows the tip of a cantilever twisted on the flat surface and on the top edge of the step grating. When friction happens on the flat surface, the moment balance equations at O point of the tip can be calculated by

$$\sum \mathbf{F}: \quad \mathbf{F} = \mathbf{F}_f + \mathbf{N}_S = -\mu N_S \mathbf{i} + N_S \mathbf{j} \tag{4}$$

$$\mathbf{R} = -H\sin\theta_0 \mathbf{i} - H\cos\theta_0 \mathbf{j} = -H\theta_0 \mathbf{i} - H\mathbf{j} \quad (\theta_0 \text{ is very small}) \tag{5}$$

$$\mathbf{M}_o = \mathbf{R}\times\mathbf{F} = -\mu H N_S - H\theta_0 N_S = k_t\theta_0 \tag{6}$$

In Eq. (4), N_s is the normal force, F_f is the frictional force, and μ is the coefficient of friction. In Eq. (5), H is the distance between the center of twist for a cantilever and the end of a tip as shown in Fig. 6 and θ_0 is the torsional angle on the flat surface of the step grating. In Eq. (6), k_t is the torsional stiffness.

On the top edge of the step grating, the moment balance equation at O' point can be calculated by

$$\begin{aligned} &\sum \mathbf{F}: \quad \mathbf{F} = \mathbf{N}_T + \mathbf{F}_T = -\left(N_T\sin\beta + \mu N_T\cos\beta\right)\mathbf{i} + \left(N_T\cos\beta - \mu N_T\sin\beta\right)\mathbf{j} \\ &\qquad\qquad where \;\; F_y = N_T\left(\cos\beta - \mu\sin\beta\right) = N_S \end{aligned} \tag{7}$$

$$\mathbf{R} = -H\sin(\alpha+\beta)\mathbf{i} - H\sin(\alpha+\beta)\mathbf{j} \tag{8}$$

$$\mathbf{M}_{O'} = \mathbf{R}\times\mathbf{F} = -HN_s\left[\sin\theta_1 + \cos\theta_1\left(\frac{\sin\beta + \mu\cos\beta}{\cos\beta - \mu\sin\beta}\right)\right] = k_t\theta_1 \tag{9}$$

In Eq. (8), α is half of the tip angle (about 30~35°), and β is the angle between the tip and the step. When a frictional test in AFM is performed, the normal force is held constant. Thus, in Eq. (7), the force in the y direction becomes the normal force. Because the torsional angle on the top edge is small and $\alpha + \beta + \theta_1$ is 90°, Eq. (9) can be simplified to Eq. (10).

$$-HN_s\left[\theta_1 + \left(\frac{\cos\alpha + \mu\sin\alpha}{\sin\alpha - \mu\cos\alpha}\right)\right] = k_t\theta_1 \tag{10}$$

Eq. (3), (6), and (10) can be transformed to Eq. (11) in which the torsional angle on the flat surface can be calculated.

$$\theta_0^2 + N_s \tan\alpha \frac{(a-1)}{a(b+N_s)}\theta_0 + \frac{N_s{}^2}{a(b+N_s)^2} = 0$$

$$\text{where } a = \frac{V_1}{V_0} \text{ ; experimental data} \tag{11}$$

$$b = \frac{k_t}{H} \text{ ; dimensional data}$$

$$N_s \quad \text{ ; input data}$$

In Eq. (11), a is a value measured from frictional tests, b is dimensional data of a given cantilever, and N_s is a constant condition in the experiments as the normal force.

C. Torsional stiffness

The torsional stiffness of a cantilever can be calculated from classical mechanics. The torsional stiffness for the cantilever of a rectangular shape is given by Eq. (12).

$$k_t = \frac{GWt^3}{3L} \tag{12}$$

In Eq. (12), G is the shear modulus and W, L and t are the width, the length and the thickness of the cantilever. The torsional stiffness for the cantilever of a V shape can be calculated from the equation induced by W. A. Neumeister and W. A. Ducker.[23] Fig. 6 details the geometry of rectangular cantilevers. In Table I, torsional stiffnesses are given for the 4 cantilevers with the dimensions.

IV. EXPERIMENTS AND RESULTS

A. Experimental preparations

The specimen is a step grating with 500nm height from MicroMasch Inc.. The step grating is coated with Si_3N_4 to prevent Si from oxidation. Rectangular cantilevers with 0.76 N/m bending stiffness from Olympus Inc. are used (k_2 in Fig. 6 and Table I). The tip radius is about 30 nm. Because the tip height is 2.9 μm and the thickness of cantilever is 0.8 μm, the distance between the center of the twist of a cantilever and the end of the tip is 3.3 μm. Experimental instrument is AFM (SPM 400) from Seiko Instrument Inc. Experiments are conducted under normal forces from 0.74 nN to 150 nN. The frictional distance is 5 μm and the frictional speed is 1 Hz (10 μm/s). Temperature and humidity are measured.

B. Results and discussions

Fig. 7 shows frictional signals on the flat surface of the step gating. In general, a frictional force is not zero for an adhesive force when the normal force is zero at the nanometer scale. It can be considered that there is little effect of the adhesive force when a normal force is larger than 50 nN. Fig. 8 indicates the maximum signals on the top edge of the step gating. The torsional angle of the cantilever is calculated by Eq. (11) with the frictional signals and the maximum signals. Fig. 9 shows the torsional angles and the frictional signals at each normal force. Because in Eq. (11), the adhesive force is not considered, the torsional angle is zero when the normal force is zero, but the frictional signal is not. Thus, when the normal force is small, the angle conversion factor is very small for a large frictional force, but when the normal force becomes larger than 50 nN, the factor converges as shown in Fig. 10. If the adhesive force is considered in Eq. (11), the angle conversion factor would be calculated as this constant value for all normal forces. Thus, in the proposed calibration method, the angle conversion factor is considered as the converged value at normal forces above 50 nN. The angle conversion factor for the AFM used in this study is chosen to be 145 μrad/V (the dashed line in Fig. 10). The force conversion factors of cantilevers in Table I can be calculated by applying this factor in Eq. (2). In Table II, it can be seen that the force conversion factors of the cantilevers are very different.

V. VERIFICATIONS

Two experiments are performed to confirm the proposed calibration method. First, a step grating with 100 nm height is used to replicate the experiment. Second, frictional behaviors on Mica are compared with different cantilevers.

A. Another step grating

The experiment is performed with a step grating of 100 nm height to calculate the angle conversion factor as the proposed method. The results are indicated in Table III. The angle conversion factor obtained from this experiment is 152 μrad/V. This factor is nearly agreement with the factor of the experiment with step grating of 500 nm height.

B. Frictional coefficients on Mica

Frictional tests on Mica are performed using cantilevers with bending stiffnesses of 0.76 N/m and 0.10 N/m shown in Table II. The frictional signals are calibrated by the force conversion factors in Table II. Fig. 11 shows the results of frictional forces after calibration. In Fig. 11, it is found that the coefficients of friction calculated by the two cantilevers are 0.086 and 0.081, values that can be considered the same as the material property. Therefore, it should be considered that the proposed calibration method can be applied to the quantitative calibration of frictional force in AFM.

VI. DISCCUSSION

In this article, a new calibration method of frictional signals has been proposed for quantitative analysis in AFM. For the calibration, experiments were performed and the equation for calculating the angle conversion factor was set up using a moment balance equation. The calibration method is verified experimentally.

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TABLE I. Dimensions and torsional stiffnesses of each cantilever

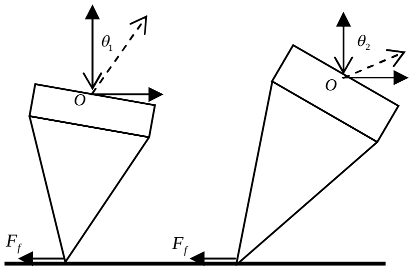
k_b (N/m)	L (μm)	W (μm)	t (μm)	k_t (nNm/rad)
$k_1 = 0.39$	100	20	0.8	1.77
$k_2 = 0.76$	100	40	0.8	3.53
$k_3 = 0.05$	200	20	0.8	0.87
$k_4 = 0.1$	200	40	0.8	1.74

TABLE II. Force conversion factors of each cantilever

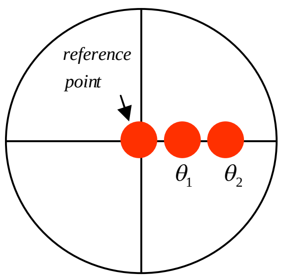
k_b (N/m)	k_t (nNm/rad)	Force Conversion Factor (nN/V)
$k_1 = 0.39$	1.77	77
$k_2 = 0.76$	3.53	155
$k_3 = 0.05$	0.87	38
$k_4 = 0.1$	1.74	75

TABLE III. Angle conversion factors calculated with the step grating of 100 nm height

Normal Force (nN)	Angle Conversion Factor (μrad/V)
50	142.5
70	146.3
90	151.4

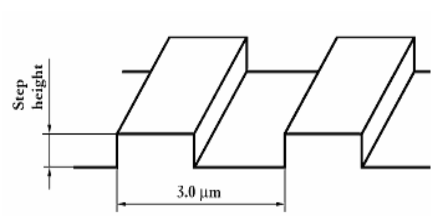


(a)

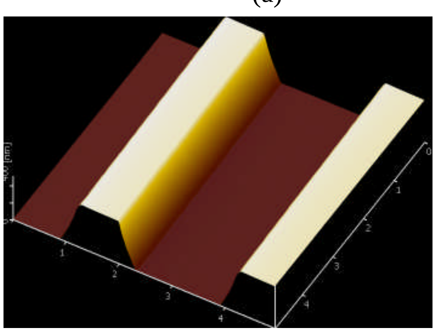


(b)

FIG. 1. (a) Two kinds of cantilevers with different tip heights make different torsional angles with the same frictional force. (b) On the detector, the laser spots move to the locations of θ_1 and θ_2 by the twist of cantilever from the reference point.

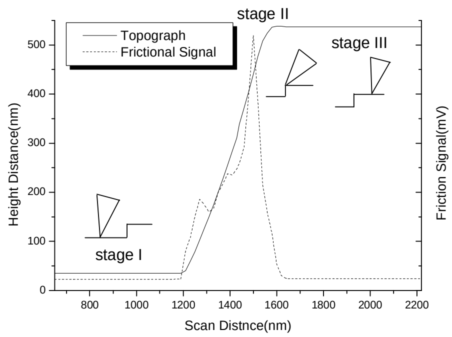


(a)

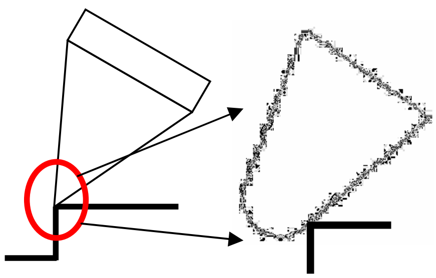


(b)

FIG. 2. (a) A schematic diagram and (b) an AFM image of a commercial step grating.



(a)



(b)

FIG. 3. (a) The frictional signal and the topography at each location. (b) The location on the tip where the maximum signal happens.

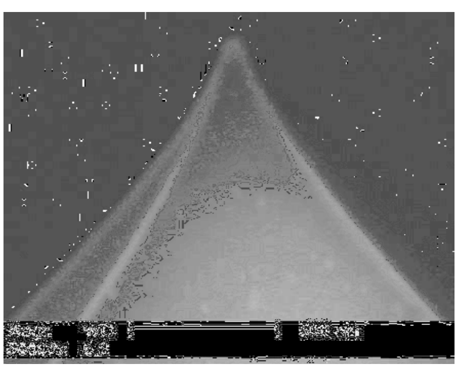


FIG. 4. The SEM image of the tip apex. The side line of the tip is not straight at the apex.

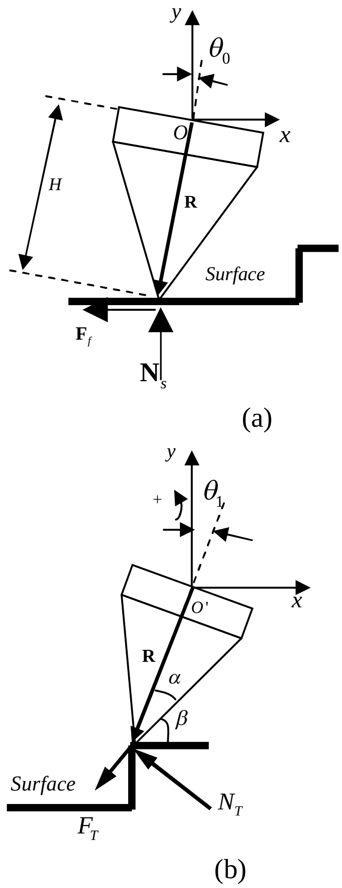


FIG. 5. The orientation of the tip for a cantilever (a) on the flat surface and (b) on the top edge of the step grating.

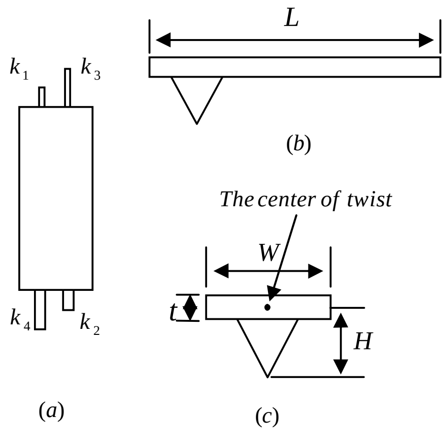


FIG. 6. Four kinds of cantilevers and geometries. (a) Silicon chip has 4 rectangular cantilevers. (b) The length of one of 4 cantilevers (c) The other geometries of a cantilever.

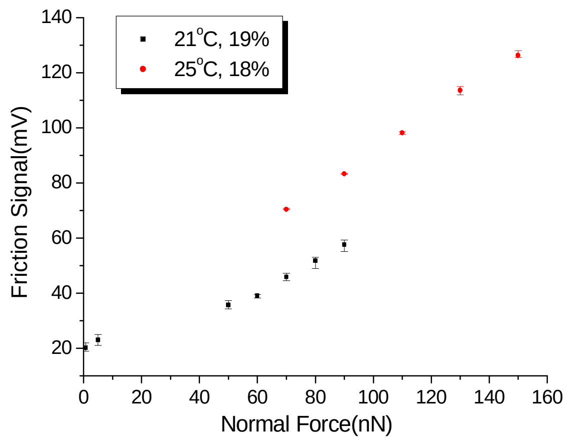


FIG. 7. Frictional signals vs. normal force. Because of different temperature and humidity in the experimental environment (i.e. different adhesive forces), the magnitude of the frictional signals is different, but the tendency of the signals (i.e. the coefficient of friction) is the same.

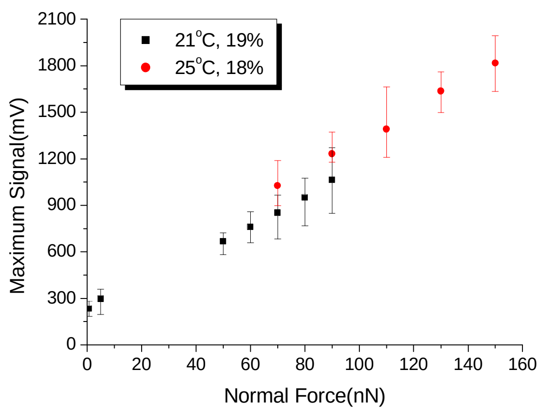


FIG. 8. The maximum signals for each normal force on the top edge of step grating.

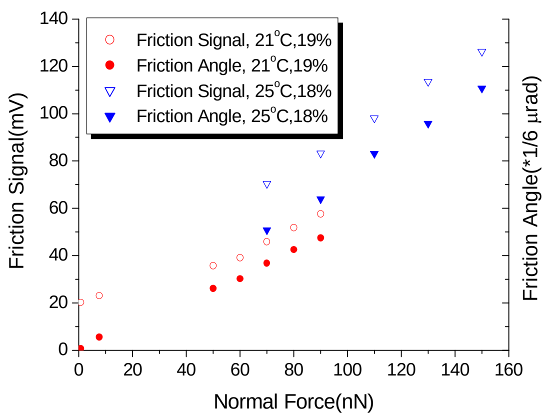


FIG. 9. The torsional angles calculated by Eq. (11) and the frictional signals at each normal force.

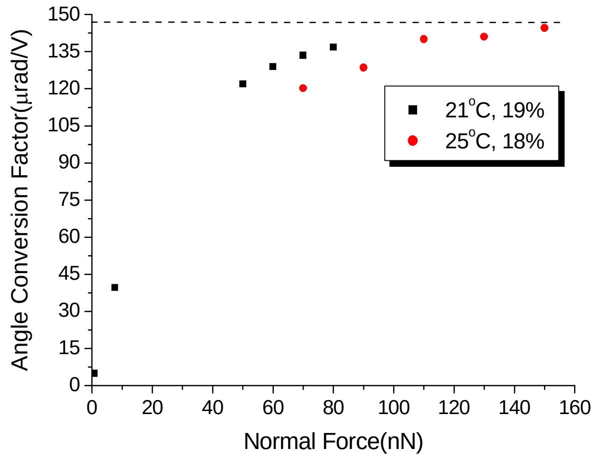


FIG. 10. The angle conversion factor at each normal force.

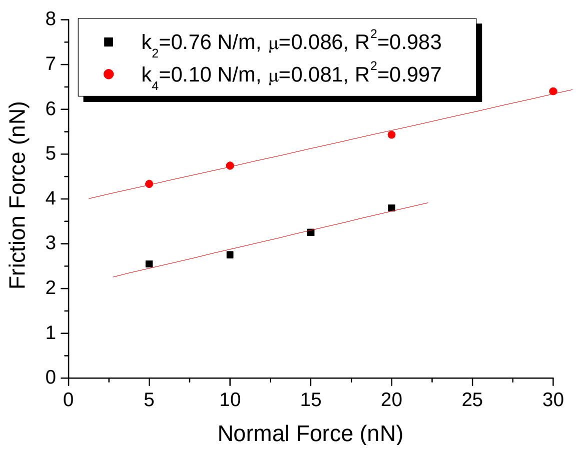


FIG. 11. The results of frictional tests performed by cantilevers with bending stiffness of 0.76 N/m and 0.10 N/m on Mica.