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A Comparison of Friction Force Calibration in Lateral Force Microscope

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Abstract: The main principle of two widely used methods which were proposed by Ruan and Bhushan, and by Ogletree and Carpick are introduced. Experiments were conducted using the two methods to measure friction force between AFM probe and silicon sample quantitatively. To characterize the frictional properties, the conversion factors of the two methods by which lateral electronic signal is converted into actual friction force were calculated. The experimental results show that that the conversion factors were extraordinarily different from each other. Further research should be done to identify the reasons for these differences.

Keywords: nano, tribology, friction, lateral force microscope, calibration

1. Introduction

Atomic Force Microscope (AFM) makes it possible to image the surface in micrometer to the subnanometer scale [1]. Additionally, the AFM is used in nanotribological applications [2]. Numerous methods [3-11] have been proposed to quantitatively determinate the micro/nano friction force between the tip of the cantilever and sample surface.

Meyer and Amer proposed a method to measure lateral force and normal force simultaneously by AFM [3]. In this method, friction force signal can be detected when AFM probe scans perpendicular to the long axis direction and friction force can be calculated quantitatively by multiplying conversion factor to the signal. Because there are so many uncertainties in spring constant or conversion factor, Ruan and Bhushan [4] came up with a method based on parallel scanning to determine friction coefficient to calculate friction coefficient without spring constant or conversion factor. In wedge method proposed by Ogletree and Carpick [5], AFM cantilever scans across two slopes of which angles makes with horizontal direction are known. The friction information can be obtained by analyzing the force variation between uphill and downhill motion. Varenberg et al. so improved the wedge method that it can be applied to common slope and colloid probe [6]. Liu applied a physical method to determine the sensitivity of optical photo detector [7]. Carpick [8] processed the tip sample contact as two serially connected springs to determine lateral shear stiffness, which is proportional to friction force. Cain [9] and Liu [10] did further researches which were also based on initial stage of friction loop and gave detail sequence to detect friction force and shear stiffness.

Although various techniques were developed as described, the methods proposed by Ogletree and Carpick (wedge method) [5] and by Ruan and Bhushan (RB method) [4] are most widely used.

In this study, to compare the two methods, fundamentals of force detection techniques in lateral force microscopy (LFM) are described. And, friction force is measured and calibrated by wedge method and RB method separately. Finally, the comparison result of the two methods is presented.

2. Experimental Details

In the experiment, lateral force measured by using a multimode SPM (MultiMode SPM III, Digital Instruments, USA). The configuration of instrument is shown in Fig. 1.

The standard silicon nitride triangular cantilever of which the quoted value of normal stiffness is 0.58 N/m was applied in our experiment. The experiment was conducted in the ambience circumstance, of which relative humidity was at 45 ± 5% and temperature at 24 ± 1°C. Silicon wafer (100) was

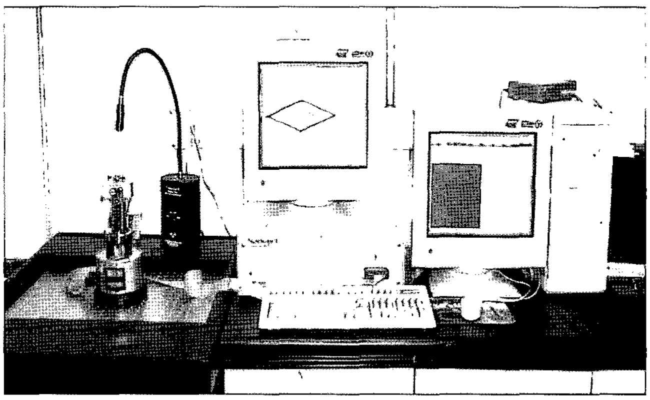

Fig. 1. Multimode Scanning Probe Microscope used in this study.

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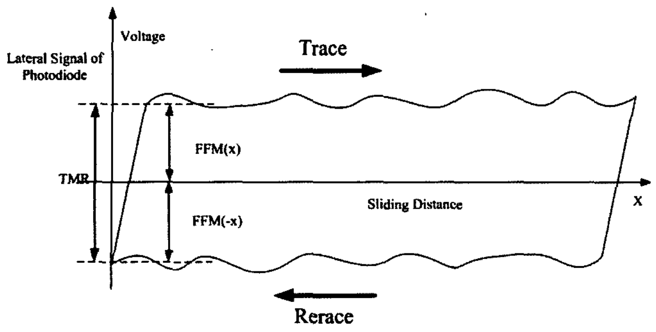


Fig. 2. Typical friction loop of lateral signal response while AFM scanning trace and retrace perpendicular to long axis of the cantilever.

used for RB method and silicon standard sample, TGF_11 (Mikromasch, USA) for wedge method.

When the cantilever scans in perpendicular to long axis of the cantilever, the lateral force will result in lateral motion of laser spot in the photodiode detector. Ideally, the cantilever's trace and retrace motions across sample surface lead to opposite electronic signal and form friction loop, as shown in Fig. 2.

The half TMR value of the friction loop represents the amplitude of the friction force. In the surfaces, which are obeyed by Amonton's First Law of friction, coefficient of friction (μ) can be obtained from the gradient of a plot of friction force (F_f) versus applied load (F_N), according to the formulae $F_f = \mu(F_N + F_A)$, where F_A is adhesion force present between two contact objects [11]. By scanning cantilever in perpendicular direction with changing normal loads and recording the corresponding lateral signal, we can get a plot of lateral friction signal TMR_{lat} with respect to normal loads.

From this, to determine friction force quantitatively, we should know exact relationship between lateral electronic signal and actual friction force to facilitate friction force detection. So, many researches are focused on determining this relationship.

3. Results and Discussion

Although many of techniques are referred to detect friction force as we mentioned above, seldom they agree with each other by our experimental validation. Here we compare two widely used techniques, Ruan and Bhushan method and Ogletree method, to get quantitative friction force and also calibrate friction force separately.

3.1. Ruan and Bhushan method (RB method)

Ruan and Bhushan proposed a method to calibrate friction by scanning a surface in the direction parallel to the long axis of the cantilever [4]. If the signal value of vertical direction from photodiode is maintained constantly during scanning in two opposite directions, the vertical reflection also keeps constant amplitude at the end of the cantilever. But the friction force acting on the tip is reversed with changing scanning direction as shown in the schematic diagram in Fig. 3.

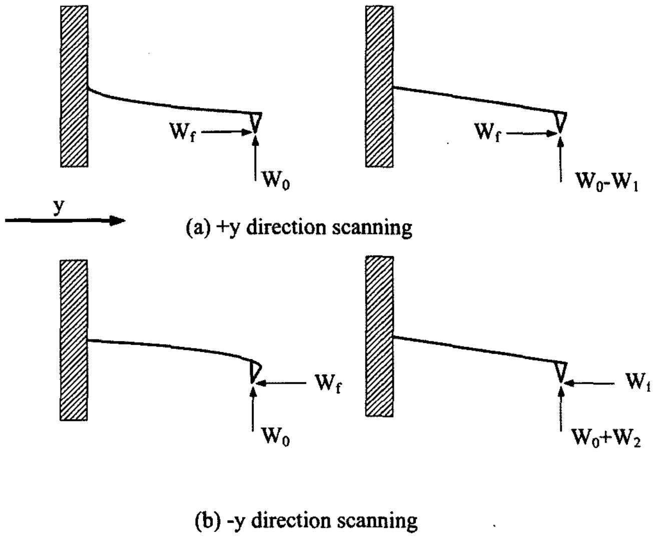


Fig. 3. Schematic diagram of cantilever bending when it scans in both (a) *y* and (b) *-y* directions.

If there is no horizontal force between the sample and the cantilever, We can assume that the force W_0 generates deflection. Since the variation of normal load is very small, the friction forces in both directions can be regarded as an equal value. In the point on which the cantilever joins the substrate, total moment acting on the point generates a corresponding vertical deflection.

This relationship yields the following equation

$$(W_0 - \Delta W_1)L + W_f l = (W_0 + \Delta W_2)L - W_f l \tag{1}$$

Where, ΔW_1 and ΔW_2 are changes of normal force W_1 and W_2. From this equation, we can get the coefficient of friction as,

$$\mu = W_f / W_0 = \frac{(\Delta W_1 + \Delta W_2)}{W_0}\frac{L}{2l} \tag{2}$$

Actual PZT displacements, ΔW_1, ΔW_2 and W_0 can be converted into detected signals, as $\Delta W_1 + \Delta W_2 = k(\Delta H_1 + \Delta H_2)$ and $W_0 = k_{nor} H_0$, where ΔW_1, ΔW_2 and W_0 correspond with vertical signal of ΔW_1, ΔW_2 and W_0, and the symbol k_{nor} presents normal spring constant of the cantilever. So, equation (2) can be rewritten as

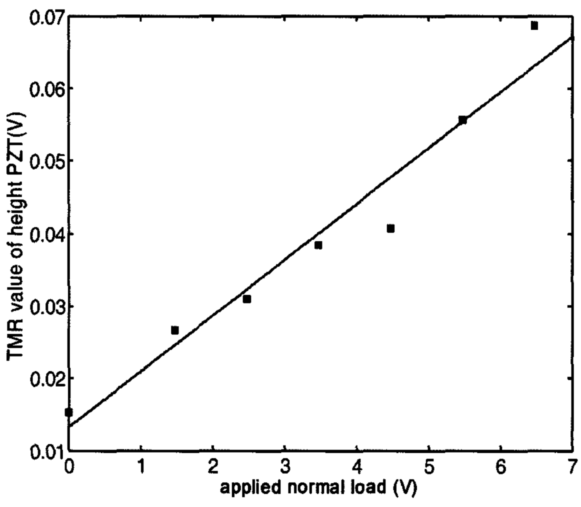
$$\mu = W_f / W_0 = \frac{(\Delta W_1 + \Delta W_2)}{H_0}\frac{L}{2l} \tag{3}$$

To eliminate the influence of adhesion force due to capillary condensation and inter-atomic force present between tip and sample surface, it is necessary to conduct experiments in a range of applied normal loads to rewritten above formulae in the differential form

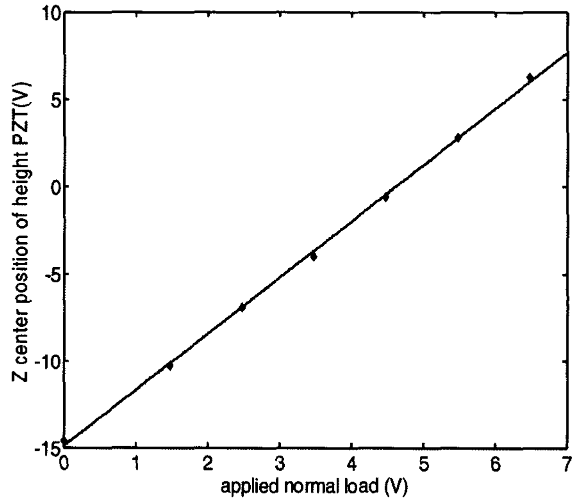
$$\mu = \frac{\Delta(\Delta H_1 + \Delta H_2)}{\Delta H_0}\frac{L}{2l} \tag{4}$$

So, the friction coefficient is obtained by conducting AFM scanning in the direction parallel to the long axis direction.

The results we got by scanning the silicon surface in the direction parallel to long axis of the cantilever is shown in Fig.



(a)



(b)

Fig. 4. plot of PZT signal with respect to applied normal load: (a) TMR value of PZT height (b) Z center position of PZT height.

4. The TMR value at each scan location is plotted in Fig. 4 (a) and slope value is 0.0082. And Z center position plot is shown in Fig. 4 (b) and the slope value is 3.2966. According to the experimental configuration, the value of $L/2l$ is 2.2578. So, the coefficient of friction for our setup is 0.00558.

Fig. 5 shows the lateral friction signal with respect to applied normal load. This signal can be obtained from parallel scanning.

The slope of the plot is 11.23 mV/V and this value means the ratio between lateral signal and normal load. If the frictional properties are constant regardless of scanning direction, by using the value, we can calibrate friction force conversion factor. In here, the conversion factor is 10.86 nN/V.

3.2. Ogletree Method

Ogletree and Carpick [5] came up with a method to conduct friction force calibration by comparing lateral signal response when the cantilever scans upwards and downwards along two

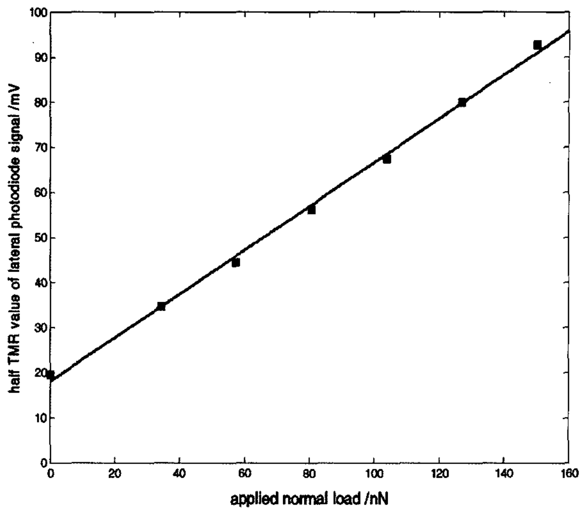
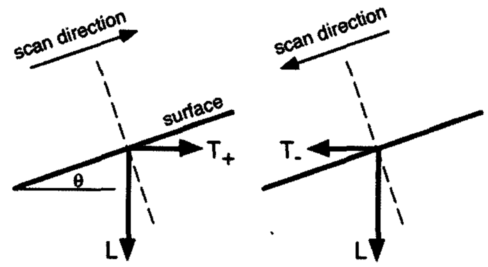
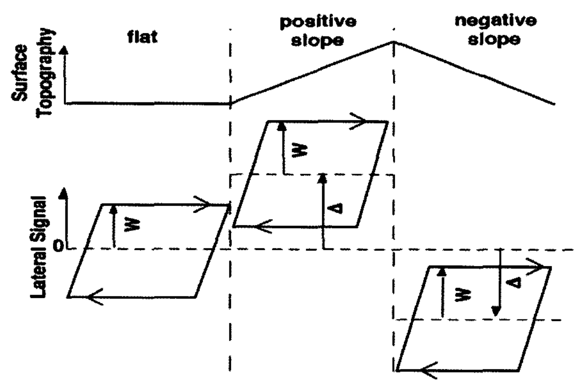


Fig. 5. Lateral friction force with respect to applied normal load.



(a)



(b)

Fig. 6. (a) Forces analysis of wedge method (b) Friction force of the motion on flat part, positive slope and negative slope.

slopes that have rigorous angle with respect to each other.

As shown in Fig. 6 (a), with a constant normal load L applied on the cantilever tip, the friction forces generated between the tip and the sample are different between uphill motion and downhill motion. From equilibrium of the forces, we can get the following equations for uphill and downhill motions separately.

$$T_+ = \frac{L\sin\theta + \mu L\cos\theta + \mu A}{\cos\theta - \mu\sin\theta} \quad (5)$$

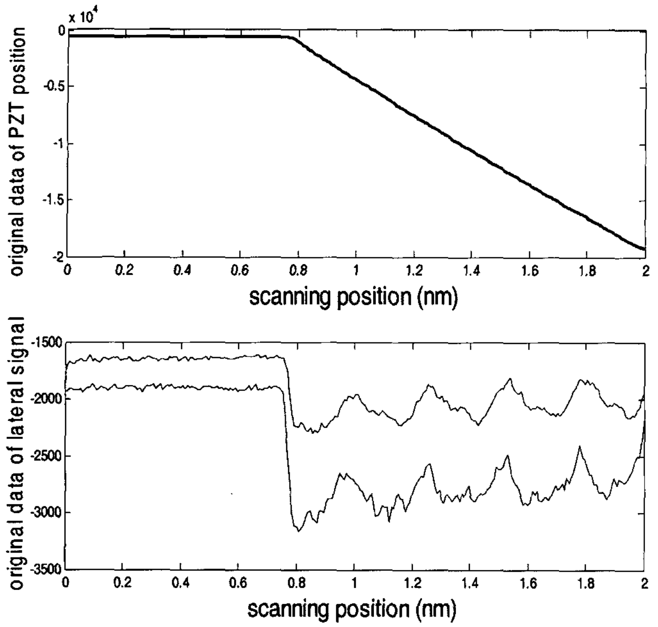


Fig. 7. Topography of the sample and corresponding lateral signal.

$$T_- = \frac{L\sin\theta - (\mu L\cos\theta + \mu A)}{\cos\theta + \mu\sin\theta} \tag{6}$$

Where, T and A represent lateral and adhesive force, respectively, μ is the coefficient of friction.

Fig. 6 (b) shows the changes of friction loop when the cantilever scans across the sample on flat, positive slope and negative slope. The variation of lateral signal corresponds to the change of lateral force.

If, F_{lat} is lateral force applied on the end of tip and V_{lat} is lateral voltage signal produced by lateral force F_{lat}, $\alpha V_{lat} = F_{lat}$ and $\beta L_0 = L$, Where α and β is conversion factors, L_0 is electronic signal of normal load and L is the actual normal force.

For actual force, we can get the following equations.

$$\alpha W_0 = W = \frac{1}{2}(T_+ - T_-) \tag{7}$$

$$\alpha \Delta_0 = \Delta = \frac{1}{2}(T_+ + T_-) \tag{8}$$

By combining equations (5) (6) and (7) (8), we can deduce the following equations in differential form

$$\alpha/\beta \cdot W'_0 = \frac{\mu}{\cos^2\theta - \mu^2\sin^2\theta} \tag{9}$$

$$\alpha/\beta \cdot \Delta'_0 = \frac{(1+\mu^2)\sin\theta\cos\theta}{\cos^2\theta - \mu^2\sin^2\theta} \tag{10}$$

We can get the slope values W'_0 and Δ'_0 by applying different normal loads L in a range during experiments. By combining equations (9) and (10), we can get the friction coefficient as,

$$\mu^2 - \frac{2\Delta'}{W'\sin2\theta}\mu + 1 = 0 \tag{11}$$

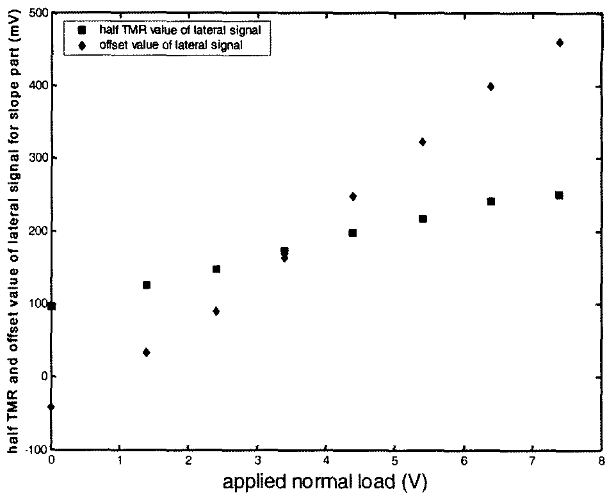


Fig. 8. Half-TMR and offset values with different applied normal load on the slope part of sample.

By resolving this equation, we can get the coefficient μ of friction.

By scanning the sample, the image shown in Fig. 7 is obtained. The top graph is the topography of the sample. The below one corresponds to the lateral signal.

The uphill signal and downhill signal were recorded to provide TMR and offset value of friction loop with respect to applied normal load, as shown in Fig. 8.

The slopes of half TMR and Offset value are 21.8094 mV/V and 70.6251 mV/V respectively. Substituting this value in the equation (7), we can get $W_0 = 21.8094$ and $\Delta_0 = 70.6251$. By resolving the equation, we get two values and select one less than 1. The coefficient we get is 0.1281. And then insert the formula $\mu = 0.1281$ into equation (5) or (6), the factor α/β can be obtained as 18.21. For our system, the value β is 23.2 nN/V. So, the conversion factor is $\alpha = 422.24$ nN/V.

3.3. Comparison of two method

Table 1 shows the friction coefficients and conversion factors from RB method and wedge method.

We find out that there are huge differences in both coefficient and conversion factor of the two methods for the same cantilever and sample. To find out the reason which causes the difference, we must get down to corresponding fundamentals or the experimental procedure which the error may be included and lead to drastic mistake.

In RB method, although we can detect the length of the cantilever, the tilting angle of cantilever relative to the line along which cantilever scans and tip height is not rigorously

Table 1. Comparison of the results obtained from RB method and wedge method

Method	Friction coefficient	Conversion factor (nN/V)
RB method	0.00558	10.86
Wedge method	0.1281	422.24

accurate. This will lead to error of the value $L/2l$. There is possibility of wrong assumption that the friction force is identical after the scanning direction changes can be satisfied only when the changes of normal load is extremely small.

In wedge method, there are some uncertainties that may lead to error signal. Ideally, the offset value of "friction loop" for flat part should be independent of applied normal load. But during experiment, we find out the value also changes with increasing normal load. This may be partly caused by so called cross talk of photodiode detector, the phenomenon that the variation of signal in normal direction of photodiode may lead to change of lateral signal due to misalignment of laser spot on the photodiode. Another reason can cause the change of offset value for flat part is the tilting caused by increasing normal load. If this is caused by "cross talk", this part of component should also be eliminated form the offset value of slope part of the sample.

Because LFM system is very sensitive and has great ambiguity, the reason of error is not yet known and much more research should be done to find out the resolution.

4. Conclusions

Two kinds of methods were applied to calibrate friction force with the same kind of the sample and cantilever. It turns out to be different in results of the two methods. Quantitative determination of micro/nano friction force is not an elementary work and needs lots of knowledge about material, cantilever and devices used in experiments. These make hard to study micro/nano friction and still, there is no commonly used method for quantitative measurement of micro/nano friction. So, much more research should be carried out to try to find the reasons.

Acknowledgments

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