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TRIBOLOGY METHODS

A New AFM Nanotribology Method Using a T-Shape Cantilever with an Off-Axis Tip for Friction Coefficient Measurement with Minimized Abbé Error

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Abstract A new AFM (atomic force microscopy) nanotribology method using a T-shape cantilever with an off-axis tip (Nat Nanotechnol 2:507–514, 2007) has been developed for measuring friction coefficient at nanometer scale. In this method, signals due to both bending and twisting of the T-shape AFM cantilever are detected simultaneously. For a T-shape AFM cantilever, the bending is caused by the normal load and the twisting is caused by both the normal and the lateral loads. The twisting generated by the normal load is calibrated in advance. Consequently, the twisting only due to the lateral load can be decoupled from the total lateral voltage signal. And the friction coefficient can be finally determined based on a conversion relationship between the normal and lateral voltage signals of the AFM photodetector. A practical procedure for minimizing Abbé error in friction coefficient measurement has also been introduced. The proposed new method is simple and accurate, and requires the least operation for friction coefficient measurement at nanometer scale.

Keywords Nanotribology · AFM · T-shape cantilever · Friction coefficient

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1 Introduction

Tribology covers fundamental research and practical applications involving friction, lubrication, and wear of interacting surfaces in relative motion, the existence of which has been witnessed everywhere in our daily life and modern industry. Appropriate utilization of tribology can reap a significant amount of economic and environmental benefits such as the reduction in use of both energy and materials. Therefore, understanding the tribological phenomena, especially the frictional mechanisms of interfaces, is of fundamental significance. With the recent trend of science and technology in making miniaturized devices at micro- and/or nano-scales [1–3], study of micro/nanotribology has become more important. To date, AFM (atomic force microscopy) has been developed as a powerful tool in lateral friction measurement [4–8], as well as for chemical mapping of surfaces at the nanometer scale [9, 10].

In literature, two major AFM methods have been proposed for friction coefficient measurements. The first one includes successive steps of calibrating the normal signal sensitivity, normal stiffness, lateral signal sensitivity, and lateral stiffness before measuring the friction coefficient. Unlike the normal sensitivity which can be directly calibrated in the force mode, the calibration of the lateral sensitivity typically requires access to the optical geometry of AFM systems [11]. However, the access to the optical path is generally unavailable on commercial AFM systems. Therefore, a number of special accessories have been designed for measuring the lateral sensitivity [12–15]. However, strict requirements for alignment tolerance increase the design complexity and related machining cost. On the other hand, calculation of the lateral stiffness requires accurate information about the cantilever geometry and high degree of material homogeneity of the cantilever [16–18]. It is difficult to obtain the information to meet the rigorous requirements on the measurement accuracy.

As a result, the second method was developed for directly determining the conversion relationship between friction force coefficient and lateral voltage response, which bypasses the aforementioned difficulties in separate measurements of the lateral sensitivity and stiffness as in the first method [9, 19, 20]. However, this direct method needs a well-defined wedge as a reference. Additional steps are required to scan both of the tilted and flat surfaces of the wedge at a given load to calculate the friction force calibration factor. Since the tilt angle of the wedge needs to be known exactly, the originally proposed calibration standard [19] used a treated $SrTiO_3$ specimen. Owing to the small dimensions of its terraces, the $SrTiO_3$ specimen is only suitable for very sharp probe. As an alternative, Tocha et al. [9] fabricated an universal calibration platform on a Si(100) wafer. The platform contains several notches with four different slopes with respect to the wafer surface. Accurate microfabrication process using focused ion beam milling was involved.

In this study, we have developed a simple AFM method by only using a T-shape cantilever with an off-axis tip for measuring friction coefficient. This new method does not require the aforementioned accessories and additional calibration steps. Thus it is easy-to-use, and needs the least operating procedures among the existing methods. In addition, a practical method has also been introduced, which can significantly reduce Abbé error.

2 Experimental Method and Results

Recent progress in micro-/nano-fabrication technology allows fabrication of T-shape optical-lever AFM cantilevers with an off-axis tip as shown in Fig. 1. T-shape AFM cantilevers are commercially available now. Sahin et al. have developed the so-called HarmoniX® mode by tapping such T-shape cantilever on a polymer sample, in which force–distance curves per tapping cycle are reconstructed and analyzed in real time for compositional mapping of Young's modulus, adhesion, and dissipation of the sample [21]. This type of cantilever has been extensively applied on torsional harmonic mode for mapping materials properties at nanoscale [22–24].

We report here that, for the first time, a T-shape cantilever is applied in contact mode for fast friction coefficient measurement due to its unique T-shape design. The typical radius of the off-axis tip of a T-shape cantilever is less than 10 nm, which ensures that our measurement is in nano-asperity contact. In the contact mode, the change in normal voltage signal ΔV_n on the AFM photodetector is proportional to the normal loading force F_n by [9, 11, 15]:

$$F_n = k_c S_n \Delta V_n \tag{1}$$

where S_n is the deflection sensitivity and k_c is the normal stiffness of the cantilever used in this article, nominally ~4 N/m (HMX—10, Veeco Inc.). Both of the sensitivity and the stiffness can be accurately calibrated in experiments if the normal force needs to be known [25]. However, to be discussed in the following part, our technique saves the effort to calibrate this normal stiffness for friction coefficient measurement. In addition, although this kind of cantilever was originally designed for tapping HarmoniX mode, such stiffness is much smaller than conventional tapping cantilever with typical stiffness ~40 N/m. Therefore, they can be successfully applied in contact mode for many materials [7, 9, 26, 27].

Due to the position of the off-axis tip, the normal load F_n induces a torque that twists the cantilever as shown in Fig. 2, and the resultant change in the lateral voltage signal ΔV_{Ln} detected by the AFM photodetector is

$$\Delta V_{Ln} = C_0 F_n = C_0 k_c S_n \Delta V_n = C_0' \Delta V_n \tag{2}$$

where both C_0 and C_0' are coefficients. Only the coefficient C_0' is required and can be easily calibrated in advance as shown in Fig. 3, where the normal voltage ΔV_n increases as the increasing normal load while without commencing scan (scan size = 0 nm or the tip is not moving). C_0' is nearly

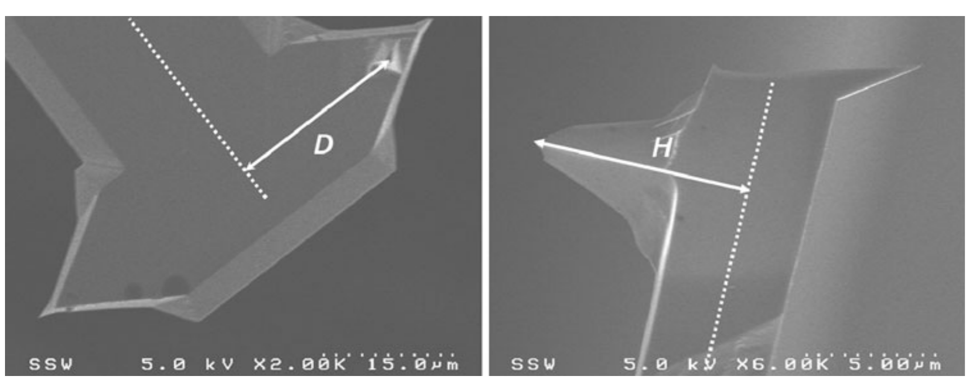

Fig. 1 Scanning electron microscopic (SEM) images of a T-shaped cantilever whose tip height (H) of 8.45 μm and tip offset (D) of 21.12 μm have been characterized

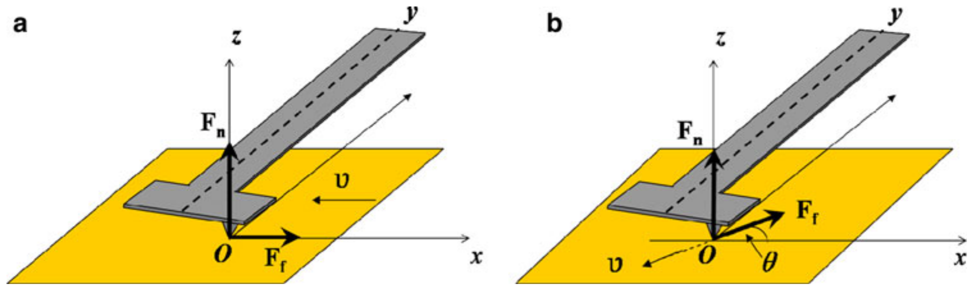

Fig. 2 Schematic illustration of the principle of using T-shape cantilever for measuring friction coefficient in contact mode. *Left* **a** perfect alignment between the cantilever width axis and the scanning line; *Right* **b** Abbé error sin θ involved due to misalignment between the cantilever width axis and the scanning line

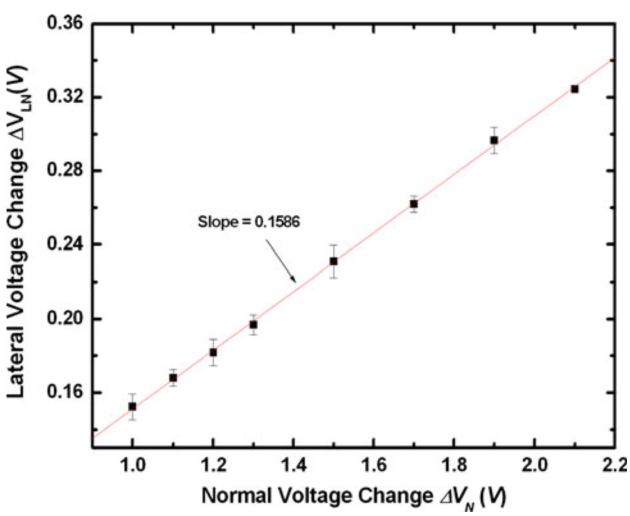

Fig. 3 Calibration of the coefficient C_0'according to the linear relationship between the resultant lateral voltage signal ΔV_{Ln} and the normal voltage ΔV_{n} when an increasing normal load applied to the tip without scanning

independent of the substrate in calibration, but affected by laser spot size, spot location, and effective power received by the photodetector [28, 29]. Recently, the increasing applications of friction force microscopy for studying frictional properties of single cells [30] or organic thin films [31] in liquid also require re-calibration of C_0' since, in liquid, the coefficient C_0' is further affected by refractive indices of media along the optical path [29, 32, 33]. Therefore, the coefficient C_0' should be always re-calibrated when the cantilever and/or the operation conditions are changed.

Furthermore, it should be noted that, during the calibration, the tip tends to move sideways if the normal load is too big. In order to avoid this problem, in practice, one should make sure the collected calibration data are in good linearity. Only in this way can the calibration be valid, for example, as shown in Fig. 3.

Once commencing the lateral scan, under the normal force F_{n} there is a friction forceF_{f} between the tip and the sample. Both F_{n} and F_{f} can cause twisting of the T-shape cantilever, and hence cause a change of lateral voltage signal on the photodetector. However, the change of the lateral voltage due to F_{n} can be decoupled and offset from the total lateral voltage. Therefore, we are able to detect the lateral voltage change due to F_{f}, ΔV_{Lf}, only. The lateral voltage change due to F_{f} can be expressed as

$$\Delta V_{\mathrm{Lf}} = C_1 F_{\mathrm{f}} = C_0 F_{\mathrm{n}}' \tag{3}$$

where F_{n}' is an equivalent normal force of F_{f} assuming that F_{n}' causes the same torsional moment as F_{f} about the cantilever longitudinal axis. They can be correlated as

$$F_{\mathrm{n}}' = \frac{F_{\mathrm{f}} H}{D}. \tag{4}$$

Therefore,

$$F_{\mathrm{f}} = \frac{\Delta V_{\mathrm{Lf}} D}{C_0 H} \tag{5}$$

where H is the height of the tip and D is the distance from the tip to the longitudinal axis of the cantilever as depicted in the scanning electron microscopy (SEM) images shown in Fig. 1. SEM is a convenient tool in precisely measuring these two geometrical parameters of the T-shaped cantilever. For the cantilever shown in Fig. 1, H and D were estimated to be 8.45 and 21.12 μm, respectively. The SEM we used was a Hitachi S-4500, which was calibrated using a standard for dimension measurements. Since the resolution of SEM is usually several nanometers, the characterization of the micrometer-scale H and D of a cantilever is relatively simple and fast. For example, it took less than half an hour to obtain the images shown in Fig. 1. For commercially available AFM probes, specifications such as the cantilever thickness and the tip height are provided. Based on our measurements, most of nominal specifications are fairly accurate. However the tip height, H, can vary to a substantial extent. For example, according to the manufacturer (Veeco Inc.), the tip height, H, of the T-shaped cantilever we used here is specified within the range of 4–10 μm. D is directly specified by the manufacturer, but can be indirectly calculated by other geometrical specifications. It is recommended to calibrate the dimensions such as H and D in order to ensure accurate measurement of friction coefficient in this method. SEM appears to be the most practical tool to calibrate cantilever dimensions.

Finally, the friction coefficient can be expressed as [19]:

$$\mu = \frac{F_f}{F_n} = \frac{\Delta V_{Lf}}{C_0 F_n}\frac{D}{H} = \frac{\Delta V_{Lf} D}{C_0 k_c S_n \Delta V_n H} = \frac{\Delta V_{Lf}}{C_0' \Delta V_n}\frac{D}{H}. \quad (6)$$

As a result, our method only needs to detect the normal voltage signal $\Delta V_n$and the lateral voltage signal related to F_f, ΔV_{Lf}, for measuring the friction coefficient. As shown in Fig. 4, the measured slopes $\frac{\Delta V_{Lf}}{\Delta V_n}$on polystyrene (PS), freshly cleaved mica and HOPG are 0.02484, 0.02359, and 0.0003469 in ambient environment, respectively. The corresponding friction coefficients according to Eq. 6 are listed in Table 1. These values fall correspondingly in the ranges of friction coefficients reported in literature for these samples [34–38] as shown in Table 1 for comparison.

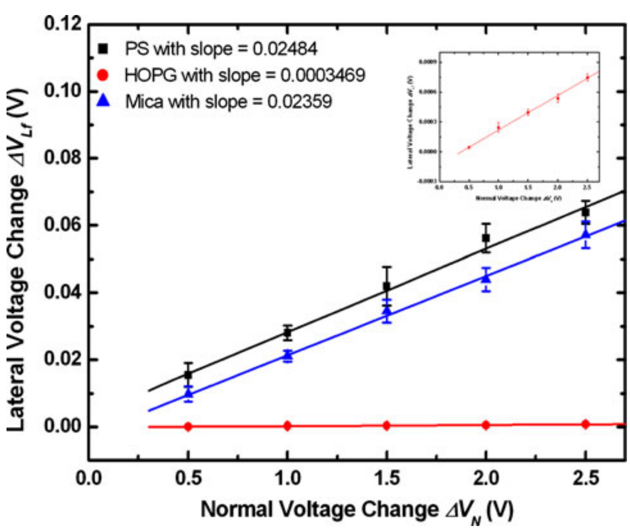

Fig. 4 The relationship between the lateral voltage signal related to F_f, ΔV_{Lf}, and the normal voltage signal ΔV_n when increasing the normal load with scan size 1 μm and scan rate 1 Hz on samples of polystyrene, freshly cleaved mica and freshly cleaved HOPG in ambient air with humidity 50% and at temperature 22 °C

Table 1 Comparison of friction coefficients measured by our T-shaped cantilever method and conventional AFM methods

Sample	T-shape cantilever method	Conventional AFM method(s)
PS	0.391	0.33–0.50 [32], 0.458 [37]
Fresh cleaved mica	0.371	~0.33 [36], 0.05–0.3 [35]
HOPG	0.005	~0.006 [34]

We, therefore, confirm the validation of the proposed method for friction coefficient measurement.

Specially, a softer T-shaped cantilever with stiffness ~0.18 N/m was customized by Dong et al. [39]. Using such softer T-shaped AFM cantilevers, our technique could be extended to analyze much softer samples including self-assembled monolayers [40] and living cells [41] in contact mode.

3 Discussion of Sources of Experimental Error

Abbé error, which is often encountered in friction coefficient measurement, is engendered by misalignment between measuring straight line (x-axis) and scanning straight line [42]. Herein, we provide a solution to minimize its effect.

First, we define a Cartesian reference coordinate O-xyz: x-axis and y-axis are along the width direction and the length or longitudinal direction of the cantilever, respectively, and they are located in the scanning x–y plane; z-axis is normal to the scanning plane (see Fig. 2). Ideally, we expect that the lateral scanning line is in perfect coincidence with the x-axis. However, when loading the cantilever on the probe holder, there is always misalignment between the cantilever longitudinal axis and y-axis. As a result, Abbé error, $\sin\theta$, is introduced and the y-axis component of friction force causes a longitudinal buckling on the cantilever and the resulted voltage signal V_{Vn} on photodetector is given by [36]:

$$V_{Vn} = C_1 F_f \sin\theta \quad (7)$$

where C_1 is a coefficient, and θ is the Abbé (misalignment) angle.

Such buckling results in additional deflection of the cantilever and introduces additional normal voltage signal on V_n. It causes an error in measurement of friction coefficient. Nevertheless, based on Eq. 7, if we can adjust the scanning angle in x–y plane to compensate the Abbé angle θ, there is a possibility to reduce the related effect. As demonstrated in Fig. 5, when the tip scans with perfect alignment $\theta = 90°$, there is no friction loop on the normal voltage signal, and both of trace and retrace scan lines overlap; in other misalignment cases such as $\theta = 0°$, 89°, or 92° friction loops clearly appear, where the upper curve is due to positive buckling and lower curve is due to negative buckling. Through this no-friction-loop method, we can effectively minimize the Abbé error within 1° in our experiments. Our AFM system can only allow the least increment or decrement of scanning angle by 1°, thus 1° is the limit for the minimization of the Abbé angle.

Our method relies on the use of the T-shape cantilever in contact mode. For a traditional cantilever, there is

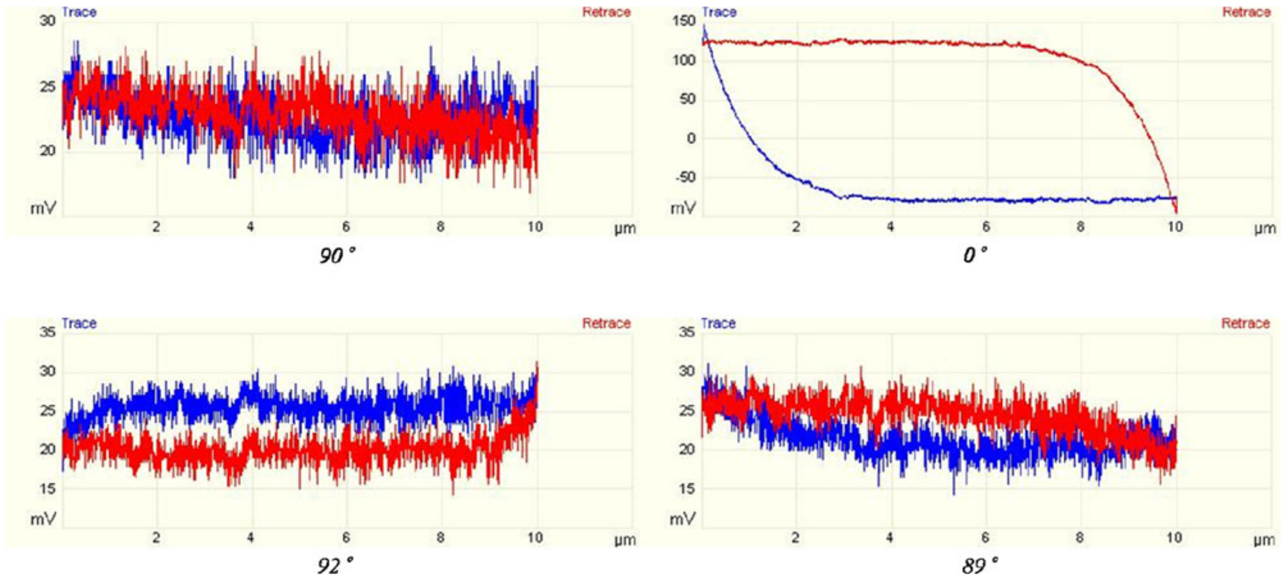

Fig. 5 Normal voltage signals of the AFM photodetector at different θ with lateral scanning size 10 μm on sapphire. *Top left* $\theta = 90°$; *Top right* $\theta = 0°$; *Bottom Left* $\theta = 92°$; *Bottom right* $\theta = 89°$

always, more or less, a tip offset D due to microfabrication defects. However, for traditional cantilevers, the uncertainty in measuring D using SEM probably is at the same order of the value of D, which will cause large deviation if we use Eq. 6. In contrast, the uncertainty in measuring D of a T-shape cantilever is much less than the value of D. Thus, T-shape cantilever design with deliberated tip offset improves the experimental accuracy in this method. In friction coefficient measurement, experimental errors may come from scan size, noise level, and sample roughness.

4 Conclusions

In summary, we have developed a simple and accurate AFM method for fast friction coefficient measurement in contact mode. This method takes advantage of a T-shape cantilever with an off-axis tip AFM, and eliminates necessities of complex and rigorous calibration procedures in advance and/or any accessories during experimental preparation. Additionally, we have also introduced the no-friction-loop method for minimizing Abbé error. This work has enriched the AFM methodology for nanotribology research.

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