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AFM lateral force calibration for an integrated probe using a calibration grating

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ABSTRACT

Atomic force microscopy (AFM) friction measurements on hard and soft materials remain a challenge due to the difficulties associated with accurately calibrating the cantilever for lateral force measurement. One of the most widely accepted lateral force calibration methods is the wedge method. This method is often used in a simplified format but in so doing sacrifices accuracy. In the present work, we have further developed the wedge method to provide a lateral force calibration method for integrated AFM probes that is easy to use without compromising accuracy and reliability. Raw friction calibration data are collected from a single scan image by continuous ramping of the set point as the facets of a standard grating are scanned. These data are analysed to yield an accurate lateral force conversion/calibration factor that is not influenced by adhesion forces or load deviation. By demonstrating this new calibration method, we illustrate its reliability, speed and ease of execution. This method makes accessible reliable boundary lubrication studies on adhesive and heterogeneous surfaces that require spatial resolution of frictional forces.

Keywords: Lateral force, friction, calibration, atomic force microscopy, calibration grating

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1. Introduction

In recent years, there has been growing interest in measuring the frictional properties of hard materials, heterogeneous polymers, and thin films [1-7]. This interest is driven by the need to characterise the nanomechanical properties of a large variety of systems with applications in biomedical engineering, biotechnology and micro- and nano- electronics [8]. Atomic force microscopy (AFM) is one of the most promising tools used for the investigation of local surface friction because of its high force sensitivity [9-11]. In a typical AFM friction measurement, an AFM tip in contact with a surface is scanned across the sample and the friction between the sample and the tip results in a detected voltage. From this voltage, frictional properties of the material can be calculated using a conversion (calibration) factor as a calibration that links the measured signal with the lateral force exerted on the cantilever by frictional drag [12]. The conversion factor depends on the lateral sensitivity of the position-sensitive photodiode, which provides a measure of the deflection of the AFM cantilever, and the cantilever's torsional spring constant [13].

Over the last 15 years, a variety of methods for calibrating lateral forces in AFM have been demonstrated [12, 14-34]. They can be loosely grouped into two groups, distinguished by their approach to lateral force calibration. In the first approach, the lateral sensitivity of the position-sensitive photodiode and the torsional spring constant of the cantilever are calibrated separately [16]. There are a number of ways to obtain the lateral sensitivity [8]. For example, the position-sensitive photodiode can be calibrated by rotating the AFM head through a range of tilt angles while the lateral voltage is measured [15], or the raw thermal noise spectrum of an AFM cantilever can be measured [35]. The torsional spring constant can be calculated using, for example, continuum elasticity mechanics of isotropic solids and measured dimensions of the

cantilever and tip [8]. However, it has been demonstrated that it is difficult to perform the calibration accurately in both the above steps due to inaccuracies in the experiments and approximations used in the calculation of the spring constant [8].

In the second, more commonly taken approach, a conversion factor is obtained without separate measurement of the lateral sensitivity of the position-sensitive photodiode or the torsional spring constant [12, 19, 27]. One of the more accepted methods for obtaining the lateral force conversion factor is the wedge calibration method developed by Ogletree et al. [12]. In this method, the calibration factor is obtained by analysing the lateral response of the AFM cantilever when scanned across a faceted SrTiO_3 surface that is specially prepared to have two well-defined slopes. However, the application of this method is somewhat cumbersome due to the requirement of this specially prepared substrate. Additionally, relatively complex calculations are needed to extract the calibration factor from the raw data. Varenberg et al. [19] simplified this method so that a commercially available trapezoidal calibration grating can be used instead of the specially prepared SrTiO_3 surface. The calibration factor obtained using this method is, however, load-dependent.

Following the method of Varenberg et al., Tocha et al. [36] calibrated cantilevers on a self-fabricated calibration specimen with tilt angles that are different to commercially available trapezoidal calibration gratings. Tocha et al. demonstrated that the direct wedge method is effective for nanotribological measurements on a wide range of geometrically different materials. The simplifications however, lead to a load dependence of the calibration factor.

Systematic errors are typically problematic when measuring lateral forces using the AFM. For example, the effects of adhesion forces are sometimes ignored, even though they contribute

significantly to lateral forces, and so must be carefully considered when calibrating and measuring friction [27]. Another assumption often made is that the load exerted on a sloped facet of the calibration grating during trace and retrace scans are the same. However, when using most commercially available AFMs, the feedback system of the AFM often leads to differences in trace and retrace loads when scanning sloped facets [37]. An important contribution made by Varenberg et al. [19] is the cancelation of some systematic errors associated with lateral force calibration. More recently, Ling et al. [27] identified a group of error sources related to raw lateral signals. These include errors caused by optical interference, poor electrical feedback, and non-zero lateral cantilever deflection even if no torque is applied to the cantilever.

In the present study, we have taken further the method of Varenberg et al. [19] and utilised the errors identified by Ling et al. [27] to develop a simple, accurate and reliable single scan method of calibrating the lateral forces of integrated AFM tips. In our method, an integrated AFM tip is scanned only once across the facets of a standard trapezoidal calibration grating, minimising the risk of tip and/or surface wear. We have eliminated the influences of adhesion and load deviation, and minimized signal errors on the lateral force conversion factor. Below we describe in detail the operating procedures, the processes of extracting and analysing the data and the removal of error sources. It is an easy-to-follow procedure for lateral force calibration that will facilitate the use of integrated AFM tips in boundary lubrication measurements to enable the measurement of spatial variations in friction on nanometer length scales and on adhesive or non-adhesive heterogeneous surfaces.

2. Experimental section

All AFM experiments were performed using an MFP-3D AFM (Asylum Research, Santa Barbara, CA). AFM cantilevers/probe chips (CSC38/no Al) and a commercial trapezoidal calibration grating (TGF11) were purchased from MikroMasch, Tallinn, Estonia. The angle of the sloped-facets of the grating is $54^{\circ}44'$ (54.7°). Rectangular cantilevers (cantilever A on the probe chip) with nominal spring constants of 0.08 N/m were used in all experiments. The nominal thickness of the cantilever is 1 μm and the nominal tip height is 22.5 μm . The MFP-3D uses a low coherence light source that minimizes optical interference.

A force curve was collected on a flat, rigid surface, and the region where the tip is not in contact with the surface (the baseline of the force curve), was zeroed using MFP-3D software (Version 080501+0410) to correct the response of the photodetector displacement in the z-direction, i.e. perpendicular to the surface. Photodetector sensitivity to normal forces was measured from the slope of the constant compliance region of a force curve taken on application of a loading force to a flat, rigid surface. Spring constants of cantilevers under normal force were determined from the power spectral density of thermal noise fluctuations in air by MFP-3D software.

A friction loop was measured by tracking the lateral voltage signal while scanning the probe over the surface at a scan angle of 90° , for both trace and retrace. The feedback system was carefully regulated by adjusting the gain values and scan rate to ensure the tip traces out the contours of the sample surface as precisely as possible. In a friction measurement, height images, deflection images and friction signal images in both the trace and retrace directions were collected simultaneously. All operations were performed in air under a relative humidity of $\sim 20\%$ and at a temperature of $\sim 25^{\circ}\text{C}$. Codes developed in-house using Matlab (Version

R2010a, Mathworks) and Igor pro (Version 6.04, Wavemetrics Inc.) were used for data analysis.

3. Results and discussion

Through the resolution of forces on and exerted by the tip, we have extended the work of Varenberg et al. [19] to develop a new method by which the lateral force conversion factor, α , and the coefficient of friction, μ , can be obtained accurately. Our method makes no assumptions about adhesion forces or applied load and eliminates a number of systematic errors. We illustrate the principles and efficacy of our method by taking the reader through the analysis details of friction scans across a standard trapezoidal TGF 11 grating.

3.1. Lateral force conversion factor from lateral force deflection measurements

As detailed above, lateral force is measured by scanning (trace and retrace) the AFM tip across a sample of interest in a direction orthogonal to the long axis of the cantilever, when the tip and sample are in contact (Fig.1a). This gives rise to torsion on the cantilever in a direction opposite to the scan direction, detected as a lateral voltage signal, V_{lateral} , from which the lateral force, F_{lateral} , of the material can be calculated, viz:

$$F_{\text{lateral}} = \alpha V_{\text{lateral}} \quad (1)$$

where α the lateral force conversion (calibration) factor. For a smooth, flat surface, the frictional force, f , is equal to F_{lateral} . Varenberg et al. [19] and Ling et al. [27] have shown that α can be obtained by measuring a friction loop on a standard trapezoidal calibration grating with facets of angle θ . A friction loop is measured by tracking the lateral voltage signal while scanning the probe over the surface at a scan angle of 90° , for both trace and retrace (Fig. 1b).

The half width, $W_0(M, \theta)$, and offset, $\Delta_0(M, \theta)$ of a friction loop are functions of torsional moment, M , and the angle of the facet being scanned, θ .

The calculation of α from V_{lateral} relies on relationships that are based on the resolution of forces acting on and exerted by the AFM probe. When scanning uphill along a facet (Fig. 2a), the forces exerted on the tip by the surface are the normal contact load, N_u , the frictional force, f_u , and the effective adhesion between the tip and the sample, F_A . These must be balanced by the applied load, F_{load_u} , the lateral force, F_{lateral_u} , and the torsional moment, M_u , exerted by the cantilever of thickness t . Similarly, the acting forces and torsional moment are also in balance for a downhill scan (Fig. 2b). Based on the resolution of forces when scanning uphill (denoted by subscript u) and downhill (denoted by subscript d), and the definition of a friction loop, the following equations are obtained:

$$F_{\text{lateral}_u} = \frac{F_{\text{load}_u} \sin \theta + \mu(F_{\text{load}_u} \cos \theta + F_A)}{\cos \theta - \mu \sin \theta} \quad (2)$$

$$F_{\text{lateral}_d} = \frac{F_{\text{load}_d} \sin \theta - \mu(F_{\text{load}_d} \cos \theta + F_A)}{\cos \theta + \mu \sin \theta} \quad (3)$$

$$M_u = F_{\text{lateral}_u} \left(h + \frac{t}{2} \right) \quad (4)$$

$$M_d = F_{\text{lateral}_d} \left(h + \frac{t}{2} \right) \quad (5)$$

$$W_0(M, \theta) = \frac{M_u - M_d}{2\beta} \quad (6)$$

$$\Delta_0(M, \theta) = \frac{M_u + M_d}{2\beta} \quad (7)$$

where μ is the coefficient of friction and α is related to β via:

$$\alpha = \frac{\beta}{h + t/2} \quad (8)$$

Using Eqns. (1) – (8) and assuming that F_{load_u} and F_{load_d} are the same (i.e. F_{load}) and equal to the set point (i.e. the force preset for a particular scan), Varenberg et al. [19] derived the following equations for calculating the lateral force conversion factor, α , for an integrated AFM tip:

$$\left(\frac{\beta}{h + t/2} \right) W_0(M, \theta) = \frac{\mu(F_{\text{load}} + F_A \cos \theta)}{\cos^2 \theta - \mu^2 \sin^2 \theta} \quad (9)$$

and

$$\left(\frac{\beta}{h + t/2} \right) \Delta_0(M, \theta) = \frac{\mu^2 \sin \theta (F_{\text{load}} \cos \theta + F_A) + F_{\text{load}} \sin \theta \cos \theta}{\cos^2 \theta - \mu^2 \sin^2 \theta} \quad (10)$$

For most commercially available AFMs, F_{load_u} and F_{load_d} are not necessarily equal to each other while scanning sloped facets, even if the feedback system has been carefully adjusted. In Varenberg et al. also assume that the static pull off force is equal to the effective adhesion force, F_A [19]. As stated above, the measured pull off force is usually not equal to the effective (dynamic) adhesion force [27]. During sliding, not all points of contact are simultaneously broken during pull off, as is the case during pull off under static (non-sliding) conditions. This was demonstrated by Johnson [38] who showed that, under the same normal load, the contact area between an AFM tip and a solid surface under static conditions is different to that when sliding. These two assumptions might be the reason Varenberg et al. observed a load dependence of the lateral force conversion factor, α [19].

In our work presented here, we avoid these approximations made by Varenberg et al. We take into account possible sources of error in the lateral voltage signal when measuring a friction loop and eliminate the contribution of adhesion and load deviation to the lateral force conversion factor. This is detailed below.

First we identify a number of error sources that can contribute to the measured half width, $W_0(M, \theta)$, and offset, $\Delta_0(M, \theta)$, of the lateral voltage signal in a friction loop. As suggested by Ling et al.[27], errors arise due to static coupling between lateral and vertical deflection voltage signals, resulting in non-zero lateral cantilever deflection even if no torque is applied. The associated errors in half width and offset are W_0^a and Δ_0^a , respectively. There can also be variation of the lateral voltage signal associated with poor electrical feedback that results in the failure of the AFM tip to trace out the contour of the surface exactly (W_0^b and Δ_0^b), and optical interference effects that lead to signal noise (W_0^c and Δ_0^c). Taking these systematic errors as additive quantities, we can relate the measured half width, $W_0(M, \theta)$, and offset, $\Delta_0(M, \theta)$, to the corresponding corrected values, $W_0^0(M, \theta)$ and $\Delta_0^0(M, \theta)$:

$$W_0(M, \theta) = W_0^0(M, \theta) + W_0^a + W_0^b + W_0^c \quad (11)$$

$$\Delta_0(M, \theta) = \Delta_0^0(M, \theta) + \Delta_0^a + \Delta_0^b + \Delta_0^c \quad (12)$$

To accurately obtain α , $\square\square\square$ corrected half width, $W_0^0(M, \theta)$, and corrected offset, $\Delta_0^0(M, \theta)$, should be used, rather than their measured values, since these contain systematic errors. These errors must therefore be removed. It is possible to achieve this experimentally. In section 3.3 below, we describe experimental procedures for doing so. Once done, the corrected values of half width and offset, $W_0^0(M, \theta)$ and $\Delta_0^0(M, \theta)$, can be used instead of $W_0(M, \theta)$ and $\Delta_0(M, \theta)$ in Eqs. (6) and (7), respectively. We can then substitute Eqs. (2) – (5) into Eqs. (6) and (7), to finally obtain:

$$\left(\frac{\beta}{h + t/2} \right) W_0^0(M, \theta) = \frac{\mu}{2} \frac{(F_{\text{load}_u} + F_{\text{load}_d} + 2F_A \cos \theta)}{\cos^2 \theta - \mu^2 \sin^2 \theta} + \frac{(1 + \mu^2)}{2} \frac{(F_{\text{load}_u} - F_{\text{load}_d}) \sin \theta \cos \theta}{\cos^2 \theta - \mu^2 \sin^2 \theta} \quad (13)$$

$$\left(\frac{\beta}{h + t/2}\right) \Delta_0^0(M, \theta) = \frac{1}{2} \left(\frac{(\mu^2 + 1)(F_{\text{load}_u} + F_{\text{load}_d}) \sin \theta \cos \theta + 2\mu^2 F_A \sin \theta + \mu(F_{\text{load}_u} - F_{\text{load}_d})}{\cos^2 \theta - \mu^2 \sin^2 \theta} \right) \quad (14)$$

In our experiments, we found that F_{load_u} is always larger than the preset load controlled through the set point (i.e. F_{load} used by Varenberg et al.), while F_{load_d} is always smaller than the preset load in the same fast scan line. We can correct for the discrepancy by using a factor termed the load deviation, ΔF_{load} , (see section 3.2), viz:

$$\begin{aligned} F_{\text{load}_u} - \Delta F_{\text{load}} &= F_{\text{load}} \\ F_{\text{load}_d} + \Delta F_{\text{load}} &= F_{\text{load}} \end{aligned} \quad (15) \quad (16)$$

It is important to note that ΔF_{load} is load-independent. Substituting Eqs. (15) and (16) into Eqs. (13) and (14) yields:

$$\left(\frac{\beta}{h + t/2}\right) W_0^0(M, \theta) = \frac{\mu F_{\text{load}}}{\cos^2 \theta - \mu^2 \sin^2 \theta} + \frac{\mu F_A \cos \theta + (1 + \mu^2) \Delta F_{\text{load}} \sin \theta \cos \theta}{\cos^2 \theta - \mu^2 \sin^2 \theta} \quad (17)$$

$$\left(\frac{\beta}{h + t/2}\right) \Delta_0^0(M, \theta) = \frac{F_{\text{load}} (\mu^2 + 1) \sin \theta \cos \theta}{\cos^2 \theta - \mu^2 \sin^2 \theta} + \frac{\mu \Delta F_{\text{load}} + \mu^2 F_A \sin \theta}{\cos^2 \theta - \mu^2 \sin^2 \theta} \quad (18)$$

Equations (17) and (18) can be rearranged to obtain expressions for $W_0^0(M, \theta)$ and $\Delta_0^0(M, \theta)$:

$$W_0^0(M, \theta) = S_W F_{\text{load}} + I_W \quad (19)$$

$$\Delta_0^0(M, \theta) = S_\Delta F_{\text{load}} + I_\Delta \quad (20)$$

where

$$S_w = \frac{h+t/2}{\beta} \frac{\mu}{\cos^2\theta - \mu^2\sin^2\theta} \quad (21)$$

$$I_w = \frac{h+t/2}{\beta} \frac{\mu F_A \cos\theta + \Delta F_{\text{load}} (1+u^2) \sin\theta \cos\theta}{\cos^2\theta - \mu^2\sin^2\theta} \quad (22)$$

$$S_\Delta = \frac{h+t/2}{\beta} \frac{\mu^2 \sin\theta \cos\theta + \sin\theta \cos\theta}{\cos^2\theta - \mu^2\sin^2\theta} \quad (23)$$

$$I_\Delta = \frac{h+t/2}{\beta} \frac{\mu^2 F_A \sin\theta + \mu \Delta F_{\text{load}}}{\cos^2\theta - \mu^2\sin^2\theta} \quad (24)$$

Eqs. (19) and (20) are linear relationships, so from plots of $W_0^0(M, \theta)$ and $\Delta_0^0(M, \theta)$ as functions of the preset loading force, F_{load} , the respective gradients, S_w and S_Δ , can be used to calculate β and the coefficient of friction, μ . The lateral force conversion factor, α can be obtained from β via Eq. (8). Since F_A and ΔF_{load} are contained in only the y-intercepts (I_w and I_Δ), they do not contribute to α or μ .

In practice, a series of friction loops are measured for a range of different applied loads, over the region of linear compliance of the cantilever, i.e. when the deflection of the cantilever in contact is proportional to piezo movement in the z-direction. By measuring on the sloped and flat facets of a standard calibration grating under optimal scan rates and gains, the measured half widths and offsets, $W_0(M, \theta)$ and $\Delta_0(M, \theta)$, can be corrected to obtain $W_0^0(M, \theta)$ and $\Delta_0^0(M, \theta)$ for the calculation of β and μ . Our procedures for this are detailed in the following section.

3.2. Methodology for lateral force calibration

To illustrate our method for lateral force calibration of an integrated AFM tip, lateral force measurements were performed on a standard TGF11 grating. Fig. 3a is a three-dimensional

AFM image of the topography of the calibration grating showing that the surface is clean, smooth and regularly faceted. The slope of the incline between peaks and troughs is well defined and consistent across the sample (Fig. 3b). The fast scan direction is set orthogonal to the cantilever axis, while the TGF11 grating is accurately mounted in the scanner to ensure it is level and that its sloped facets are parallel to the cantilever axis. Friction loops are collected to obtain $W_0(M, \theta)$ and $\Delta_0(M, \theta)$ on all facets of the standard trapezoidal calibration grating, i.e. at angles $0, \theta$ and $-\theta$.

Typically, friction loops are obtained by collecting a series of scans of the surface at different applied loads. We have simplified this procedure so that only a single surface scan is required. In our method, the set point is stepwise-adjusted at the start of each scan line to obtain a series of friction loops at different loads, within a single surface scan. We achieve the automatic set point adjustment using code from Asylum Research Technical Support modified in-house. Using this method, all information, including height images, deflection images and friction signal images, can be stored into a single image file, significantly reducing the recording time. This procedure also minimizes the risk of surface wear of both the grating and AFM tip, which can lead to incorrect calibration.

The loads applied to the surface during friction measurement can be obtained from the deflection image from which the actual deflection value can be extracted. Fig. 4a shows a deflection image of a standard trapezoidal calibration grating obtained using our single-scan method, when scanning from bottom to top. The graduation of colours across the image corresponds to the change in vertical deflection from scan line to scan line. Note that the deflection image is comprised of 256 different scan lines, each corresponding to an individual set point. The image clearly shows that, within the same scan line, the vertical deflection

signals on the flat and sloped facets are different, indicating that the load exerted on a flat facet is less than on an uphill facet, but greater than on a downhill facet.

To obtain the actual loading force applied to a surface during friction measurement, cantilever deflection must be known. In general, the actual cantilever deflection, d , is the value of deflection obtained from the deflection image plus a correction factor, which is the deflection corresponding to the set point at the end of a complete scan [37]. Fig. 4b is an example of corrected deflection, d , along two different scan lines. It can be clearly seen that d increases with an increase in preset load. Additionally, corrected deflection is different by an amount Δd on sloped compared to flat facets, along the same fast scan line. Note from Fig. 4b that Δd is approximately independent of set point for any particular image. Now, when scanning a flat surface, the actual deflection is equal to the preset deflection. This allows calculation of the preset load, F_{load} , for any fast scan line, as required for lateral force calibration when using Eqs. (19) and (20), viz: $F_{load} = k \times d$, where k is the normal force constant of the cantilever [39]. Likewise, the load deviation, ΔF_{load} , obtained when scanning sloped facets is calculated from Δd , the difference in deflection between flat and sloped facets, i.e. $\Delta F_{load} = k \times \Delta d$. Clearly, ΔF_{load} is load independent in the same scan, which validates the assumptions used to obtain Eqs. (15) and (16).

Figs. 5a and 5b show a lateral voltage image for both trace and retrace, respectively. The stepwise loading and the discrepancy in lateral signal between flat and sloped facets are clearly shown in Figs. 5a and 5b, where the lateral signal increases as the set point is increased stepwise, i.e. colour becomes brighter from bottom to top. The cross section of a scan line from the lateral voltage image gives a friction loop at a particular set point (Fig. 5c). The data show that half width and offset measured on flat facets, $W_0(M, 0)$ and $\Delta_0(M, 0)$, are very

similar across the surface, as expected. If this were not the case, it would suggest that the AFM laser and/or the position-sensitive photodetector are misaligned [40].

From friction loops and preset loads determined as described above, plots of $W_0(M, \theta)$ and $\Delta_0(M, \theta)$ as a function of preset load can be constructed. An example is shown in Fig. 6a. Note there is a clear linear dependence of $W_0(M, \theta)$ and $\Delta_0(M, \theta)$ on preset load for each of these examples and indeed for all measurements (data not shown). This indicates that for the surface and range of applied loads used here, friction between the tip and the surface of the grating follows Amontons' law ($f = \mu N$), which is known to hold for only multi-asperity contacts [8, 27]. Frictional contact with a single-asperity is more likely to follow a power law, such as the Johnson-Kendall-Roberts (JKR) theory or the Derjaguin-Muller-Toporov (DMT) model [8, 27, 41, 42].

3.3. Correcting $W_0(M, \theta)$ and $\Delta_0(M, \theta)$ for systematic errors

S_W and S_Δ , in Eqs. (19) and (20), respectively, are required to calculate the lateral force conversion factor, α , from β and the coefficient of friction, μ , using Eqs. (21) and (23). But S_W and S_Δ are obtained from the relationships between the corrected half width and offset values, $W_0^0(M, \theta)$, $\Delta_0^0(M, \theta)$ and preset loads, F_{load} , as detailed above. We must therefore correct the measured half widths and offsets, as follows.

To minimise dynamic coupling between the lateral and vertical deflection voltages it is necessary to optimise the AFM operating parameters to ensure the tip traces out the contour of the grating exactly. The corresponding errors in half width and offset, W_0^b and Δ_0^b , are thereby minimised and can be safely neglected.

Optical interference effects (associated errors denoted by W_0^c and Δ_0^c) and static coupling between lateral and vertical deflection voltages (associated errors denoted by W_0^a and Δ_0^a) must also be considered. Optical interference is negated if the AFM optics are correctly aligned. This is tested by measuring the voltage signals on flat facets at different heights. If these are the same, W_0^c and Δ_0^c are negligible [40]. Static coupling is independent of the scan direction [27]. Therefore when measuring a friction loop, W_0^a is zero since the errors in half width on trace and retrace cancel out. So since we can minimise both W_0^b and W_0^c , and W_0^a cancels out in a full friction loop measurement, we ensure that $W_0(M, \theta) = W_0^0(M, \theta)$, i.e. the measured half width needs no further correction.

Errors in the offset due to static coupling and optical interference, Δ_0^a and Δ_0^c respectively, do not, however, cancel out when measuring a friction loop. But they can be evaluated by measuring a friction loop on a flat facet of a calibration grid [27], as first suggested by Varenberg et al. [19]. Since the offset on a flat facet should be zero, any measured offset is the total error. This is then easily subtracted from the measured offset during a friction measurement to obtain the corrected value, $\Delta_0^0(M, \theta)$.

3.4. Calculating the lateral force conversion factor and the coefficient of friction from the lateral force calibration data

With the half width and offset of the friction loop corrected as described above, $W_0^0(M, \theta)$ and $\Delta_0^0(M, \theta)$ as a function of preset load can be plotted (Fig. 6b). The gradients are S_W and S_Δ , respectively. From these, $(h+t/2)/\beta$ and μ can be obtained by solving Eqs. (21) and (23) simultaneously. According to Eq. (8), the lateral force conversion factor, α , is the reciprocal of $(h+t/2)/\beta$. For the sample data in Fig. 6b, S_W and S_Δ are 0.84 and 1.59 mV/nN, respectively.

The angle of the sloped facet of the calibration grating, θ , is 54.7° . These give $(h+t/2)/\beta = 0.91$ mV.nN^{-1} , $\alpha = 1.1$ nN.mV^{-1} , and $\mu = 0.27$. Note that we do not require the dimensions of the cantilever to obtain α . The friction coefficient, μ , is within the range expected for integrated probes and TGF11 calibration gratings, i.e., $\sim 0.26 - 0.33$ [19]. We estimate the error in α to be 10%. This value is largely due to the uncertainty associated with using the thermal tune method to obtain the normal spring constant of the cantilever [43, 44].

To test the robustness of our single scan method, we have applied the classical wedge method [12] to the same set of friction data (see Supplementary Information). We tested against the wedge method since it is widely regarded as being the most effective and most accurate method to date for lateral force calibration, and so is the benchmark in the field [8, 23, 35]. This comparison showed that α and μ obtained via the wedge method are the same as obtained using our new single scan method. This further highlights that our new method does not sacrifice accuracy for expediency. It is as accurate as the classical wedge method but much simpler to implement. This agreement between our new method and the wedge method isn't surprising since both rely on determining α from the variation with preset load of the half width and offset in measured friction loops. Note also that α is comparable to the value obtained by Varenberg et al. for rectangular cantilevers, i.e., $\sim 1.5 - 1.8$ nN.(mV)^{-1} [19]. In Table 1, we have summarised key details of our new single scan method and other recognized calibration methods. We have compared these methods on the basis of utility, errors and assumptions, and load dependence. Our new method compares very favourably with these previously reported methods. It does not suffer from the restrictions of load dependence inherent to some methods, and it is the simplest contact mode calibration method to implement and the only one that minimises errors due to wear.

We have also tested the validity of our method under ambient conditions using rectangular and V-shaped contact-mode cantilevers that vary in dimension and spring constant, and were sourced from different suppliers (Bruker and MikroMasch). Fig. S1 in Supplementary Information contains the trace and retrace friction images for each of these cantilevers for comparison. Scanning electron microscopy images of a triangular and a rectangular cantilever are shown in Fig S2 in Supplementary Information. Cantilever shape and dimensions did not affect the accuracy of the friction calibration method as judged by the very low noise in the corresponding friction images. Note however there is some when the spring constant is less than 0.03 N/m. We therefore recommend the use of cantilevers with spring constants greater than or equal to 0.03 N/m when employing our method for lateral force calibration and friction measurement. This spring constant is still suitably low for friction measurements on soft materials, so our method can be readily applied to both soft and hard materials.

Finally, the lateral force can be calculated directly from the measured lateral voltage and α : $F_{\text{lateral}} = \alpha V_{\text{lateral}}$, i.e. Eq. (1). Note that if there are systematic errors, such as drift, cross talk or laser misalignment, it is difficult to compare values of lateral force from one experiment to another, given the difficulty in exactly replicating laser alignment when changing from one sample to another and any difference in noise from day to day. To avoid this problem, we make use of the fact that the half width of a friction loop is a measure of the lateral voltage and, as described above, the errors in the half width largely cancel out in the trace and retrace of a friction loop. Therefore, if there is no direction dependence in the frictional properties of the surface being scanned, the full width of the friction loop divided by 2 gives a value, V_{lateral} , that is insensitive to instrumental and alignment errors [13].

4. Conclusions

We have demonstrated a quantitative method of lateral force calibration for an integrated AFM cantilever. The method is based on scanning a calibration grating and analysing the data obtained on a sloped facet, and the data obtained on a flat facet were used to cancel out error sources. This calibration method eliminates many systematic errors, load deviation and the effects of adhesion on friction calibration. It is easy to use compared to the many currently available methods and could be widely used for the lateral force calibration of atomic force microscopy.

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Figure captions

Fig.1. (a) Schematic representation of the lateral force measurement using a beam-deflection scanning probe and a trapezoidal calibration grating. Lateral scanning of the calibration grating results in torsion of the cantilever, detected as the lateral voltage signal by a position-sensitive photodetector (PSPD). (b) A schematic representation of a friction loop along the same scan line showing the trace (red) and retrace (blue) lateral voltage. $W_0(M, \theta)$ and $\Delta_0(M, \theta)$ are the half width and offset of the friction loop, respectively, and are both functions of torsional moment, M , and the angle of the facet being scanned, θ .

Fig. 2. Schematic diagram (not to scale) showing the interaction between an AFM tip and a calibration grating with a local slope of angle θ , during uphill (a) and downhill (b) lateral scanning. The AFM tip has dimensions of height, h ; radius of curvature, R ; and thickness, t . In the uphill scanning (a), the forces exerted on the tip at the point of contact on the slope are the contact load, N_u ; the frictional force, f_u ; and the adhesion force, F_A . These are balanced by the applied load F_{load_u} ; the lateral force, $F_{lateral_u}$; and the torsional moment, M_u ; exerted by the cantilever on the tip. The forces and torsional moment involved during downhill lateral scanning, (b), are similarly balanced.

Fig. 3. (a) Three-dimensional AFM height image ($15 \times 15 \mu m^2$) of a TGF11 grating in the trace direction. (b) Cross-sectional profile of the grating taken along the red line in (a).

Fig. 4. (a) Three-dimensional image ($15 \times 15 \mu m^2$) of the raw, vertical deflection signal in the trace direction of a TGF11 grating. The image was taken in the same area as shown in Fig. 3a with the set point continuously varied, as indicated by the colour gradation. (b) Calculated

actual deflection as a function of lateral displacement. The black data are taken along the black line in (a) and the red data are taken along the red line in (a). Δd is the deviation between deflection on sloped facets compared to flat facets, along the same fast scan line. Δd is set point independent in the same image. In this example, Δd is approximately 160 nm. Note instrument parameters can be adjusted to minimise Δd , but is difficult to eliminate completely on most commercially available AFM instruments.

Fig. 5. Lateral voltage signals collected on the flat and sloped facets of a TGF 11 grating in (a) trace, and (b) retrace directions. Images are $15 \times 6.5 \mu\text{m}^2$ and were taken with a stepwise increased set point from bottom to top of each image, indicated by the increase in image brightness. (c) Friction loop showing lateral voltage as a function of lateral displacement. Trace (red line) and retrace (blue line) correspond to the fast scan lines (horizontal lines) in (a) and (b). The midpoints between the average voltages on either sloped or flat facets are the half widths, $W_0(M, \theta)$ and $W_0(M, 0)$, on sloped and flat facets, respectively. $\Delta_0(M, \theta)$ and $\Delta_0(M, 0)$ are the corresponding offsets of the midpoint relative to zero voltage .

Fig. 6. (a) Half width, $W_0(M, \theta)$ and offset, $\Delta_0(M, \theta)$, for the lateral voltage trace/retrace signals, as a function of preset load. (b) Corrected half width, $W_0^0(M, \theta)$ and corrected offset, $\Delta_0^0(M, \theta)$ as a function of preset load. $W_0(M, \theta)$, $\Delta_0(M, \theta)$, $W_0^0(M, \theta)$ and $\Delta_0^0(M, \theta)$ all vary linearly with preset load, as indicated by the respective lines of best fit (solid black lines), the correlation coefficients for which are 0.95, 0.99, 0.95 and 0.97, respectively.

Table 1

The accuracy of our new single scan method of lateral force calibration compared to some other current methods

Calibration Method	Errors and assumptions	Load dependence
New single scan method: requires a scan image of a standard calibration grating.	About 10% error due to uncertainty in normal spring constant. Tip wear and systematic error sources minimised.	No
Classical wedge method [12]: requires multiple scan images across a faceted SrTiO ₃ surface.	10% reproducibility (as quoted). No consideration of tip wear.	No
Wagner et al.[35]: non-contact calibration method uses the methods of Sader et al.[20, 45] to calculate torsional spring constant optical sensitivity.	Average 36% deviation from the classical wedge method (as quoted) due to uncertainties in cantilever and tip dimensions and data fitting.	No
Varenberg et al.[19]: requires scan across a standard calibration grating.	About 10% error. Assumes pull-off force equals adhesion force. Ignores 10% uncertainty in normal spring constant	Yes
Tocha et al. [36]: same as Varenberg et al. but uses a custom-made grating.	5% accuracy but only for high loading forces ($>2-3 \times$ the mean pull-off force). Ignores 10% uncertainty in normal spring constant.	Yes

Fig. 1

Fig. 2

Fig. 3

Fig. 4

Fig. 5

Fig. 6

- ☐ We develop a simple and accurate method for lateral force calibration in AFM friction measurements
- ☐ We detail the basis of the method and illustrate how to use it and its reliability with example data
- ☐ Our method is easy, accurate and accounts for the affects of adhesion on friction measurements
- ☐ The method is applicable to integrated probes, as opposed to colloidal probes
- ☐ This allows accurate AFM friction measurements on spatially heterogeneous and dhesive surfaces

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