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Quantitative assessment of contact and non-contact lateral force calibration methods for atomic force microscopy

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ABSTRACT

Atomic Force Microscopy (AFM) has been widely used for measuring friction force at the nano-scale. However, one of the key challenges faced by AFM researchers is to calibrate an AFM system to interpret a lateral force signal as a quantifiable force. In this study, five rectangular cantilevers were used to quantitatively compare three different lateral force calibration methods to demonstrate the legitimacy and to establish confidence in the quantitative integrity of the proposed methods. The Flat-Wedge method is based on a variation of the lateral output on a surface with flat and changing slopes, the Multi-Load Pivot method is based on taking pivot measurements at several locations along the cantilever length, and the Lateral AFM Thermal-Sader method is based on determining the optical lever sensitivity from the thermal noise spectrum of the first torsional mode with a known torsional spring constant from the Sader method. The results of the calibration using the Flat-Wedge and Multi-Load Pivot methods were found to be consistent within experimental uncertainties, and the experimental uncertainties of the two methods were found to be less than 15%. However, the lateral force sensitivity determined by the Lateral AFM Thermal-Sader method was found to be 8–29% smaller than those obtained from the other two methods. This discrepancy decreased to 3–19% when the torsional mode correction factor for an ideal cantilever was used, which suggests that the torsional mode correction should be taken into account to establish confidence in Lateral AFM Thermal-Sader method.

1. Introduction

Friction force microscopy or lateral force microscopy (LFM) is a contact-mode measurement technique that is offered in most atomic force microscope (AFM) instruments as a relatively turnkey capability. However, it is challenging to calibrate an AFM system to interpret an LFM signal as a quantifiable surface force (e.g., friction force) [1]. In contrast, calibrating for and measuring normal surface forces, such as adhesion, can be implemented with an AFM in a relatively straightforward manner. In order to measure normal forces, an AFM probe is pressed against a relatively rigid surface (relative motion in Z) to produce a normal "force–distance curve" calibration in which the normal compliance slope indicates the normal optical lever sensitivity of the system, as illustrated in Fig. 1(a). In most cases, when a silicon or silicon nitride tip is pressed against a similarly stiff surface material, the compliance of the tip and the tip-surface contact is negligible compared to the compliance of the cantilever, and hence, all deflection in the measurement can be considered to occur in cantilever. Once the normal optical lever sensitivity has been determined in volts per meter, the known normal spring constant of the cantilever, in Newtons per meter, gives the force calibration of the system.

Fig. 1(b) shows an example of the LFM friction measurement, which is also referred to as a friction-loop. The tip slides in one direction and then the other, and the height of the hysteretic loop, ΔV_L, is proportional to the friction encountered between the tip and the surface. To calibrate an AFM system to take this friction measurement, the first-approximation approach would be to consider a calibration that is completely analogous to that described above for normal forces (Fig. 1(a)). That is, the relative motion in Y is imposed between the tip and surface such that the static friction between the tip and the surface causes the cantilever to twist, and thus the lateral compliance slope can be obtained as in Fig. 1(b). The known lateral spring constant of the cantilever, in Newtons per meter can be combined with the lateral optical lever sensitivity in volts per meter to then give the lateral force calibration of the system. Unfortunately, the lateral compliance slope can rarely be used to calibrate the lateral optical lever sensitivity of the system in this manner because the tip and/or tip-surface contact in most cases is not rigid enough in terms of shear relative to the lateral stiffness of the cantilever. In addition, the cantilever itself can be relatively compliant in its XY plane in many cases [2].

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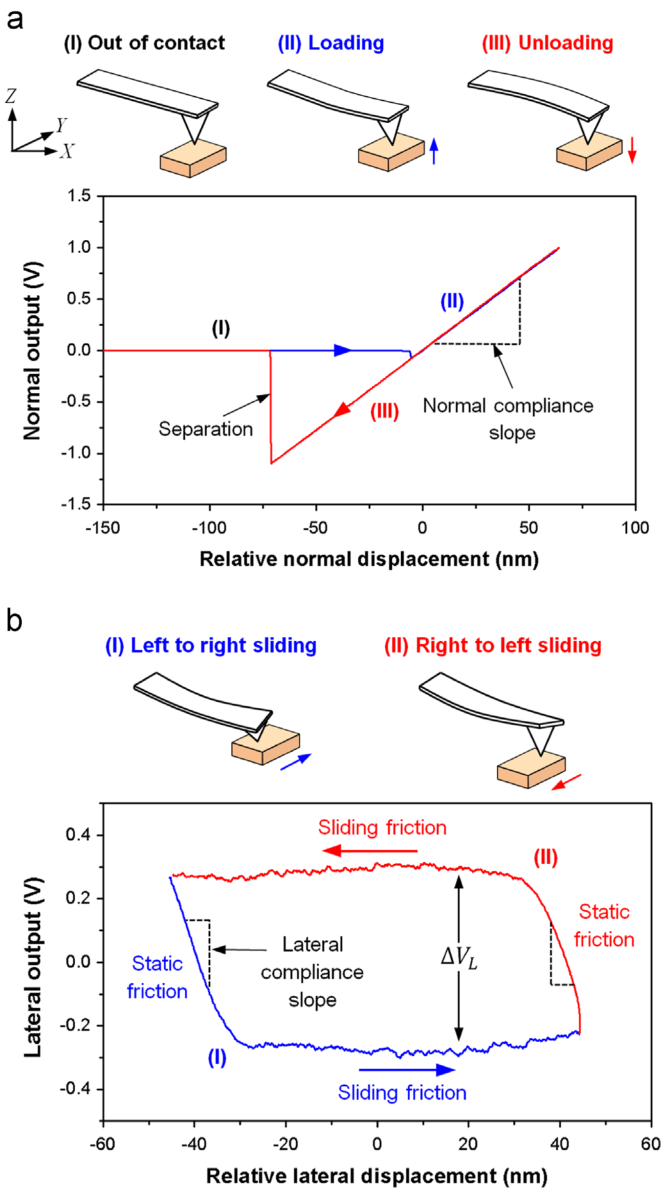

Fig. 1. Examples of (a) the normal force distance curve and (b) the friction loop along with an illustration of the AFM cantilever behavior.

Since the AFM instrument is practically sensitive only to a twist in the cantilever, the instrument senses the twist (torsion) in the cantilever (in volts) when trying to measure the lateral optical lever sensitivity in this way. However, the lateral displacement (m) is more than just the deflection due to the torsional spring of the cantilever since the other in-plane springs are deflected as well. The in-plane springs are not at all well characterized—the tip and contact compliances in particular are largely unknown—so the degree to which different springs contribute to the displacement of the lateral optical lever sensitivity measurement is also unknown. Due to these difficulties, this approach is avoided for LFM calibration of a typical cantilever with good reason, except for cases where the tip and contact compliances are negligible (e.g., a colloidal probe) [3].

While many LFM calibration methods have been proposed [1], there is a general lack of consensus and a lack of confidence in the quantitative integrity of such approaches. One key reason for this is that there is a lack of a suitable force standard that establishes the accuracy and allows test methods to be benchmarked against known quantities. A standard has been produced for normal force measurements [4,5], mostly because AFM normal force measurements are more common, but also because they are also far easier to implement (which comes back to the main problem of taking AFM lateral force measurements). Indeed several studies have been conducted with the aim to assess whether there is a quantitative agreement between two or more LFM calibration methods [6–8]. Recently, encouraging results were reported in a study comparing two physically independent LFM calibration methods that shared no critical variables to determine the final results. The study reported an agreement to within about 15% between the two methods [9]. However, it is even more challenging to produce an agreed methodology for LFM calibration due to the relative difficulty of implementing certain methods weighed against their perceived accuracy. In addition, some methods can exhibit undesirable side-effects during implementation, such as damage to the AFM tip and/or surface [1].

Considering that LFM friction measurements are themselves contact measurements where the tip slides over a surface, there are actually good reasons to consider calibration methods that minimize or eliminate contact between the tip and the surface. Due to the small size of an AFM tip, contact between the tip and the surface during either normal or lateral loading carries the real possibility of damaging the tip and/or surface [10,11]. Higgins et al. proposed a method to calibrate the normal optical lever sensitivity of AFM systems that does not require contact [12]. In addition to avoiding damage to the tip and surface during calibration, this normal “non-contact” calibration method was also developed to for use in situations where delicate materials are attached to the tip (or surface) or where the surface is possibly too soft to reliably carry out the normal optical lever sensitivity calibration. The proposed method involved the use of the Sader’s resonance method to determine the normal spring constant of the AFM cantilever [13], and this value is then used as a known parameter in the thermal noise calibration method [14–16] to obtain the dynamic (non-contact) optical lever sensitivity value of the system. Recently, Wagner et al. proposed a completely analogous calibration procedure for lateral forces that uses the Sader method to determine the torsional spring constant of the cantilever [17]. They then used this as a known parameter in an analogous thermal noise calibration of the torsional resonance of the cantilever, which is referred to as the “Lateral AFM Thermal-Sader” method [18]. A range of different cantilevers was used in their study to compare the non-contact approach to the wedge method [19] that is commonly used for LFM calibration. However, when comparing the methods, Wagner et al. encountered difficulties in implementing the wedge method and found a limited agreement between the two. In discussing the difficulties, they observed that repeated scanning of the wedge caused damage to the tips, which may have further complicated the calibration. While a commonly cited advantage of the wedge method is that it derives its lateral force calibration factor without the need to measure other factors, such as the torsional spring constant of the cantilever or the lateral deflection sensitivity of the photodiode (It does need the normal force sensitivity, as with most methods.) a criticism of this method is that the lateral force that causes the lateral response is not directly known or measured, and certain assumptions need to be made regarding the interaction of the tip with the wedge surface and how this translates to the lateral force [1]. Another disadvantage of the wedge method is its inherent susceptibility to optical crosstalk in the optical lever system. Optical crosstalk is a result of misalignment between the reflective surface of the cantilever and the sector axis of the quadrant detector that is used in LFM-capable AFM systems. In the original wedge method, the effect of the crosstalk was compensated by performing multiple wedge measurements where the differences between the relative values were used to cancel out the effects of the crosstalk.