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Fundamentals of Friction and Wear on the Nanoscale

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Fundamentals of Friction and Wear on the Nanoscale

Second Edition

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Preface

The second edition of "Fundamentals of Friction and Wear on the Nanoscale" has been motivated by the significant progress made by nano tribology in the last seven years. New chapters on triboluminescence, friction in liquids, nonlinear mechanisms of friction, fractal surfaces, multiscale modeling of contacts, capillary condensation, nano manipulation in SEM, colloidal systems, graphene, nanowear of polymers, Casimir forces, and cell motility have been added. Other key chapters, such as those on atomic-scale friction in ultra-high vacuum and nano manipulation have been completely revised. On the other side, we have omitted some chapters dealing with side aspects of nano tribology which did not undergo significant changes in the last few years. We hope that this new edition will attract the interest of a broad readership of scientists and engineers, and stimulate new experiments and theoretical models in this exciting multidisciplinary research field.

Madrid — Enrico Gnecco
Basel — Ernst Meyer

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Part IV Nanomanipulation

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Part I
Experimental Techniques

Chapter 1
Friction Force Microscopy

Roland Bennewitz

Abstract This chapter introduces Friction Force Microscopy, which is possibly the most important experimental technique in nanotribology. In spite of the apparent simplicity of this technique, a special care is required in the calibration of the force sensors, as discussed in the chapter. We will also present a few key results on the load, material and temperature dependence of friction. The chapter ends with an overview on dynamic measurements of friction, in which the probing tip is oscillated laterally while sliding in contact with the sample surface or even while translating at very close distance from it.

1.1 Introduction

Friction Force Microscopy (FFM) is a sub-field of scanning force microscopy addressing the measurement of lateral forces in small sliding contacts. In line with all scanning probe methods, the basic idea is to exploit the local interactions with a very sharp probe for obtaining microscopic information on surfaces in lateral resolution. In FFM, the apex of a sharp tip is brought into contact with a sample surface, and the lateral forces are recorded while tip and sample slide relative to each other. There are several areas of motivation to study FFM. First, the understanding of friction between sliding surfaces in general is a very complex problem due to multiple points of contact between surfaces and the importance of lubricants and third bodies in the sliding process. By reducing one surface to a single asperity, preparing a well-defined structure of the sample surface, and controlling the normal load on the contact the complexity of friction studies is greatly reduced and basic insights into the relevant processes can be obtained. Furthermore, with the decrease of the size of mechanical devices (MEMS) the friction and adhesion of small contacts becomes a technological issue. Finally, the lateral resolution allows to reveal tribological contrasts caused by material differences on heterogenous surfaces.

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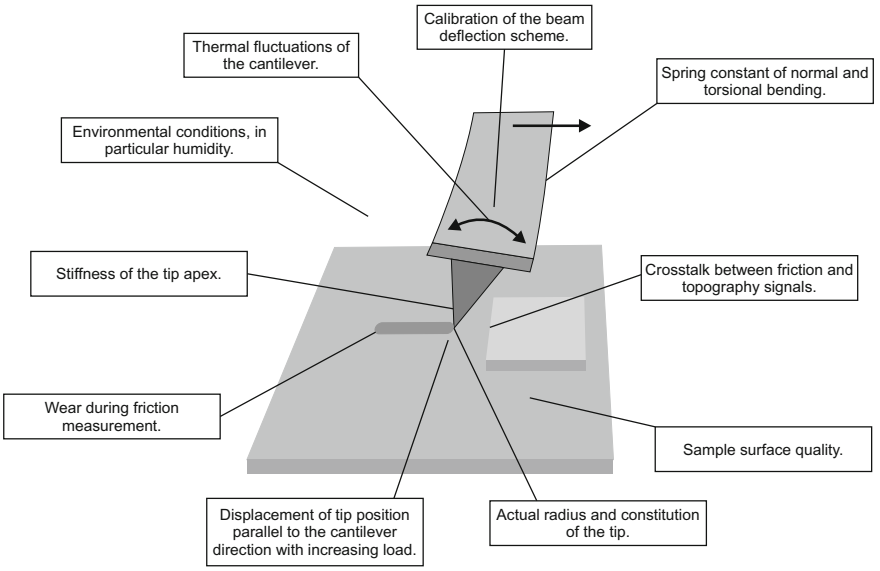

Fig. 1.1 Critical issues in experimental friction force microscopy which are discussed in this chapter

The experimental field of FFM has been pioneered by Mate et al. [1]. The group built a scanning force microscope where the lateral deflection of a tungsten wire could be measured through optical interferometry. When the etched tip of the tungsten wire slid over a graphite surface, lateral forces exhibited a modulation with the atomic periodicity of the graphite lattice. Furthermore, a essentially linear load dependence of the lateral force could be established.

In this chapter we will describe aspects of instrumentation and measurement procedures. In the course of this description, a series of critical issues in FFM will bee discussed which are summarized in Fig. 1.1.

1.2 Instrumentation

1.2.1 Force Sensors

The force sensor in the original presentation of FFM by Mate et al. was a tungsten wire [1]. Its deflection was detected by an interferometric scheme where the wire constituted one mirror of the interferometer. A similar concept was later implemented by Hirano et al., who optically detected the deflection of the tungsten wire in a Scanning Tunneling Microscope when scanning the tip in close proximity to the surface [2]. Mate and Hirano report lateral spring constants from 1.5 to 2,500 N/m, depending on the wire thickness and length. Etching the wire to form a tip at its end,

mounting the wire, aligning of the light beam, and determination of the spring constant comprise some experimental difficulties. These difficulties are greatly reduced by the use of dedicated micro-fabricated force sensors. A very sophisticated instrumental approach to the solution of those problems has been realized by Dienwiebel et al. [3]. The group has attached a stiff tungsten wire to a micro-fabricated force sensor made of silicon. The central part of the sensor is a pyramid holding the tip. The position of the pyramid is detected in all three dimensions by means of four optical interferometers directed towards the faces of the pyramid. It is suspended in four symmetric high-aspect ratio legs which serve as springs with isotropic spring constant in both lateral directions and a higher spring constant in normal direction. The symmetric design of the instrument allows for determination of normal and lateral forces acting on the tip with minimal cross talk. An overview over different experimental realizations of FFM is given in Fig. 1.2.

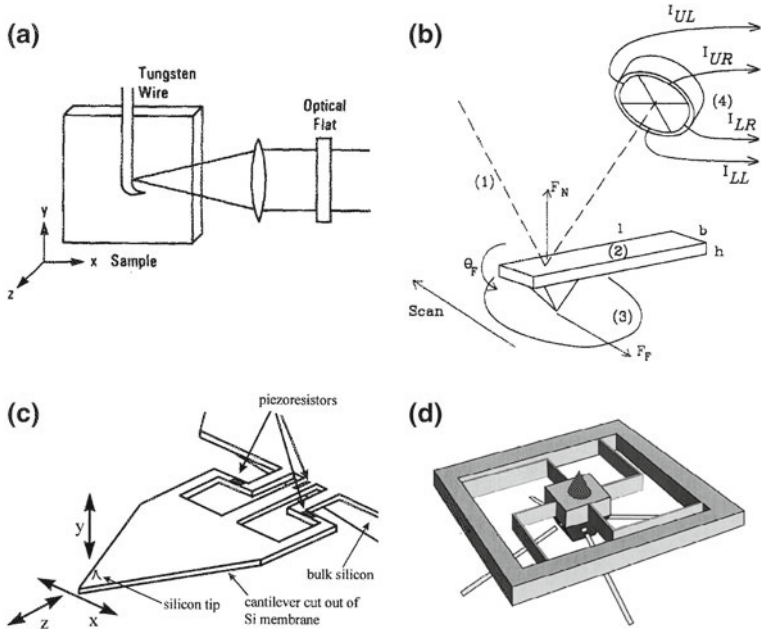

Fig. 1.2 Four design options for Friction Force Microscopy. **a** Concept of the original instrument used by Mate et al. for their pioneering experiments [1] The deflection of a tungsten wire is detected by optical interferometry. The bent end of the wire is etched into a sharp tip. **b** Beam-deflection scheme as devised by Marti et al. [5]. Normal force F_N and friction force F_F cause bending and twisting of the cantilever. The deflection of a reflected light beam is recorded by comparing currents from four sections of a photodiode. **c** Cantilever device for the measurement of lateral forces with piezoresistive detection [8]. Lateral forces acting on the tip cause a difference in stress across the piezoresistors. **d** Micro-fabricated force detector for isotropic measurements of friction forces. The block in the center holds a tungsten tip, pointing upwards in this figure. The position of the block in all three dimensions is recorded by four interferometric distance sensors which are indicated by the four light beams below the devices [9]

The most widely used form of micro-fabricated force sensors for FFM is the micro-fabricated cantilever with integrated tip. The cantilever can be either a rectangular beam or a triangular design based on two beams. The lateral force acting on the tip is detected as torsional deflection of the cantilever. This scheme has been implemented in 1990 by Meyer et al. [4] and Marti et al. [5]. It is interesting to note that the triangular design is more susceptible to deflection by lateral forces than the rectangular beam, contrary to common belief and intuition [6]. However, triangular cantilevers are less prone to the highly unwanted in-plane bending [7].

The deflection of cantilever-type force sensors is usually detected by means of a light beam reflected from the back side of the cantilever at the position of the tip. The reflected light beam is directed towards a position-sensitive photodiode which detects normal and torsional bending of the cantilever as a shift in the position of the light beam in orthogonal directions. Realistically, there is always some cross-talk between the signals for normal and torsional bending. It can be detected by exciting the cantilever to oscillate at the fundamental normal and torsional resonance and measuring the oscillation amplitude in the orthogonal channels. The cross-talk can be minimized by rotation of the position-sensitive photodiode or accounted for in the detection electronics or software. Cross-talk can transfer topographic features into the lateral force signal and create topographic artifacts from friction contrast, the latter even amplified by the feedback circuit acting on the sample height.

Calibration of the beam-deflection scheme is not a simple task, however very important in order to compare FFM results from different sources. Many publications in the past have reported on relative changes in frictional properties, without providing any calibration at all. While such relative changes certainly represent important physical findings, it is nevertheless of utmost importance to provide all experimental information available, often allowing for a rough quantitative estimate of the lateral forces. Lateral forces in FFM can easily range from piconewton to micronewton, spanning a range of very different situations in contact mechanics, and knowing at least the order of magnitude of forces helps to sort the results qualitatively into different regimes.

The calibration comprises two steps. First, the spring constant has to be determined for the force sensor. Note that the beam-deflection scheme actually determines the angular deflection of the cantilever. Nevertheless it has become custom to quantify the force constant in N/m, where the length scale refers to the lateral displacement of the tip apex relative to the unbent cantilever. Second, a relation between the deflection of the cantilever and the voltage readout of the instrument has to be established.

For the determination of the spring constant, several methods have been suggested. The easiest to calculate it from the dimensions of the cantilever. While width and thickness are easily determined by optical or electron microscopy, thickness is better deduced from the cantilever's resonance frequency. Alternatively, the spring constant can be determined from changes in the resonances caused by the addition of masses to the free end of the cantilever. Also, the analysis of a cantilever's resonance structure in air can provide the required quantities. The latter two methods have recently be described and compared by Green et al. [10]. The relation between tip displacement

and voltage readout can be established by trapping the tip in a surface structure and displacing the sample laterally by small distances. For a rough estimate one can also assume that the sensitivity of the position-sensitive photodiode is the same for normal and torsional deflection. Taking into account the geometry of the beam-deflection scheme, the torsional deflection sensitivity can be deduced from the normal deflection sensitivity (See [11] and page 352 of [12]). Since the quantification of the thermal noise driven torsional resonance can be difficult, a combination of thermal noise and beam geometry methods can be useful for the calibration of FFM [13].

A method which provides a direct calibration of the lateral force with respect to the readout voltage is the comparison with a calibrated spring standard. Recent implementations of this approach suggest as calibrated standards optical fibers [14] or micro-fabricated spring-suspended stages with spring constants that can be traced to international standards [15]. Similarly, the lateral stiffness of a magnetically levitated graphite sheet can be used as [16]. A particularly elegant method to calibrate FFM experiments is the analysis of friction loops, i.e. lateral force curves from forward and backward scans, recorded across surfaces with well-defined wedges [11, 17, 18]. Dedicated micro-fabrication design in form of a hammer-shaped cantilever can also help to calibrate the torsional bending [19].

The torsional deflection of a cantilever can in principle be detected also by optical interferometry, provided that the beam diameter is smaller than the cantilever and the point of reflection is shifted off the torsional axis [20]. However, FFM results including normal and lateral force measurements require the differential reading of multiple interferometers [3, 21].

An alternative to the detection of the cantilever bending via the beam-deflection scheme is the implementation of piezoresistive strain sensors into the cantilever. In order to measure both lateral and normal forces acting on the tip in FFM, two such strain sensors need to be realized on one sensor. Chui et al. have created a piezoresistive sensor which decouples the two degrees of freedom by attaching a normal triangular cantilever to a series of vertical ribs sensing lateral forces [22]. Gotszalk et al. have constructed a U-shaped cantilever with one piezoresistive sensor in each arm, allowing for the detection of lateral forces at the tip [23]. While the publications presenting these novel instrumental approaches contain experimental proofs of concept, no further use of piezoresistive sensors in FFM experiments has been reported. This is certainly due to a lack of commercial availability. Furthermore, the signal-to-noise ratio in static force measurements using piezoresistive cantilevers seems not to reach that of optical detection schemes.

1.2.2 Control Over the Contact

The exact knowledge of the atomic configuration in the contact between tip apex and surface is prerequisite for a complete understanding of the results in Friction Force Microscopy. It is the most severe drawback in FFM that this knowledge is not available in most cases. While sample surfaces can often be prepared with atomic

precision and cleanliness, the atomic constitution of the tip apex is usually less controlled. Friction signals vary with tip shape, as has been investigated for steps on graphite [24]. Furthermore, in the course of sliding atoms may be transferred from the tip to the surface or vice versa. Such transfer processes occur even for very gentle contact formation, as shown in experiments combining Scanning Probe Microscopy with a mass spectrometry analysis of the tip apex [25–27]. The transfer of atoms may quite often not only quantitatively but also qualitatively change the lateral forces encountered. Chemical reactions between surface and tip have been found to significantly increase friction between a Pt(111) surface for silicon but not for diamond tips [28]. The occurrence of atomic stick-slip motion can depend on the establishment of a certain degree of structural commensurability between tip and surface in the course of scanning [29, 30]. For atomic stick-slip measurements on graphite surfaces, the role of small graphite flakes attached to the tip has long been discussed and recently confirmed experimentally [1, 31].

The best control over the atomic structure of the tip apex has been achieved for metal tips in vacuum environments. By applying the established procedures of Field Ion Microscopy (FIM), the tip structure can not only be imaged but also conditioned on the atomic scale. Cross et al. have characterized the adhesion between a tungsten tip and a gold surface and proved the conservation of the atomic tip structure by means of FIM [32]. Even with instruments of lower resolution, FIM can at least be used for cleaning procedures and for a determination of the crystalline orientation of the apex cluster [2].

The integrated tips at the end of micro-fabricated silicon cantilevers have a well-defined crystalline orientation, usually pointing with the (100) direction along the tip. However, the tip surface and with it the whole tip apex are at least oxidized and possibly contaminated through packaging, transport, and handling. Furthermore, many tips are sharpened in a oxidation process which introduces large stresses at the apex. While etching in hydrofluoric acid can remove the oxide and for some time passivate silicon surface bonds by hydrogen, a stable formation and reproducible characterization comparable with FIM of metal tips has not yet been reported. Tips integrated into silicon nitride cantilevers are amorphous due to the chemical vapor deposition process and may exhibit an ever more complex structure and chemistry at the tip apex.

One way of overcoming the uncertainty of the tip constitution is to use methods of surface chemistry to functionalize the tip [33]. Specific interactions between molecules attached to the tip and molecules on the surface can be sensed by means of FFM [34]. At the same time, very strong adhesion has been reduced by covering the tip with a passivating layer to allow for lateral force imaging for example on silicon [35]. Numerous studies using this method have been published, mainly concentrating on organic monolayers on tip and surface. A review of the field has been given by Leggett et al. [36]. While most tip functionalization relies on thiol bonding to gold-coated tips, carbon bonding to nanocrystalline diamond tips has also been realized [37]. Schwarz et al. have prepared well-defined tips for FFM by deposition of carbon from residual gas molecules in a Transmission Electron Microscope, keeping control of the tip radius for a quantitative analysis of a contact mechanics

study [38]. Force measurements explicitly aiming at interactions between colloidal particles and a surface have been performed by gluing micrometer-sized spheres of the desired size to the cantilever [39, 40]. As a final note, one should always be aware of the possible occurrence of major tip wear which has been observed to happen in a concerted action of mechanical and chemical polishing [41].

1.3 Measurement Procedures

The standard measurement in FFM is the so-called friction loop: The lateral force acting on the tip is recorded for a certain distance of scanning in the direction perpendicular to the long cantilever axis and for the reverse direction. The area in the loop represents the dissipated energy, and the area divided by twice the distance is the mean lateral force. It is always very instructive to record the topography signal of forward and backward scan at the same time, as differences will reveal cross-talk between normal and torsional bending of the cantilever.

Whenever lateral forces are measured as a function of some experimental parameter, the influence of that parameter on adhesion should be studied simultaneously. In order to interpret the experimental results in terms of contact sizes versus dissipation channels the knowledge of adhesion is essential. An excellent example is the jump in lateral forces observed on a C_{60} crystal when cooling to the orientational order-disorder phase transition, which was fully explained by a change in adhesion [42]. For experiments carried out in ambient environment, the dominant contribution to adhesion are usually capillary forces which dependent greatly on the humidity and on the hydrophobicity of the surface [43]. The humidity dependence of FFM results itself can depend again on the temperature [44–46]. Consequently, an enclosure of FFM experiments for humidity control greatly enhances the reproducibility of results.

1.3.1 Friction as a Function of Load

One of the central experiments in tribology is the quantification of friction, i.e. the change of lateral force with increasing normal load on the sliding contact. One of the questions to be addressed is whether the relation between lateral and normal force is linear for FFM experiments, i.e. whether Amontons' law extends to the nanometer scale [47]. The number of FFM studies reporting lateral force as a function of load is very large, and the overall physical picture is multifaceted, to express it in a positive way. A collection of results is shown in Fig. 1.3. From a procedural point of view it is extremely important to measure the lateral forces for the full range of small normal forces until the tip jumps out of contact, usually at a negative normal force. In this way the adhesion in the system can be categorized and even maps of adhesion can be produced from friction versus load experiments [48]. Furthermore,