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Quantitative force microscopy from a dynamic point of view

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ABSTRACT

We discuss the physical origin and measurement of force between an atomic force microscope tip and a soft material surface. Quasi-static and dynamic measurements are contrasted and similarities are revealed by analyzing the dynamics in the frequency domain. Various dynamic methods using single and multiple excitation frequencies are described. Tuned multifrequency lockin detection with one reference oscillation gives a great deal of information from which one can reconstruct the tip–surface interaction. Intermodulation in a weakly perturbed high Q resonance enables the measurement of a new type of dynamic force curve, offering a physically intuitive way to visualize both elastic and viscous forces.

1. Tip-surface force

The atomic force microscope (AFM) is the most widely used member of the family of scanning probe microscopes. Its success stems from the tremendous range of different material surfaces that it can explore, and the wide variety of different environments in which it can operate. The AFM creates an image with nanometer scale resolution, of any type of physical interaction between a sharp tip and a surface, as long as that interaction gives rise to a force between the two. For many users the AFM's primary function is mapping surface topography, but its use as an analytic microscope is becoming more widely appreciated as new methods to measure the tip-surface force with greater speed and accuracy have become available. This article will discuss various modes of AFM force measurement, with particular emphasis on the more recently developed dynamic methods and their emerging use in understanding the mechanical response of a soft material interface.

Quantitative nano-mechanics with the AFM typically refers to 'modulus mapping', where the applied force is thought to be balanced by elastic compression of two homogeneous bodies in contact. Heinrich Hertz formulated a theory in 1882 [1] which is still widely used today in the analysis of AFM data. Explaining AFM contact forces in terms of volumetric elastic compression is however problematical because surface force, which originates from interfacial energy and curvature, will eventually dominate over volumetric force as the size of the contact is reduced [2,3].

Surface forces should therefore play an essential role when an AFM tip contacts a soft material (see Fig. 1). A soft material may appear to be more fluid-like at the nanometer scale, when elastic stress becomes negligible in comparison to entropic forces as the tip penetrates into the material. Capillarity will play a larger role as the soft material can more easily deform around the sharp tip, forming an interface with very large curvature due to the very small radius of the tip ~10 nm. Some models attempt to account for both van der Waals forces, which are almost always attractive, and Hertzian contact forces in a piece-wise fashion. Others take interfacial energy into account in terms of loading curves that are not easily adapted to the measurements made with the AFM. For a review of various models see [4]. Whatever model is used, a surface map of the model parameters is often made by fitting the model's force–displacement relation to an AFM measurement that is interpreted as the quasi-static cantilever force vs. tip position curve.

Especially with soft materials one should be cautious with such an interpretation. Rather, AFM practitioners should focus their attention on the measured cantilever deflection and ask: What exactly are the forces acting on the tip and how do they depend on both cantilever deflection and cantilever velocity? In this regard dynamic methods of measuring force offer new ability to reveal the otherwise hidden information about what is really happening in the AFM contact. With dynamic force measurement we must take a more rheological view of the interaction, where the interaction force is understood to be the result of both the elastic and viscous nature of the interacting materials.

In the following we give a brief review of AFM force measurement. The discussion focuses on a particular multifrequency dynamic method of force measurement, from which we introduce the concept

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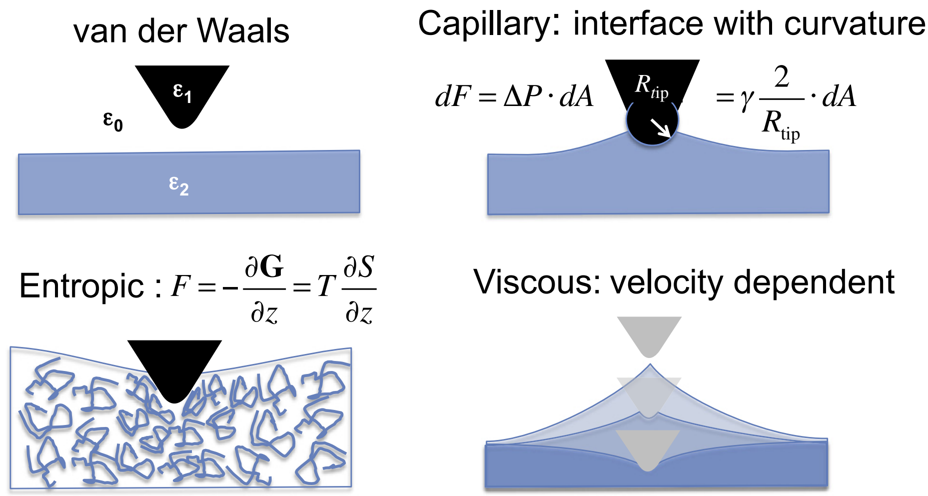

Fig. 1. A pictorial representation of different types of forces between an AFM tip and a soft sample surface: van der Walls forces due to the different dielectric permittivity ε of the tip, sample and intermediate medium. Entropic forces arising from the change in free energy G when the tip penetrates into the sample. Capillary forces resulting from the Laplace pressure jump under the tip $\Delta P = \gamma C$, given by the product of the tip-sample interfacial energy γ, and the surface curvature $C = 2/R_{\text{tip}}$. Viscous forces are present when the measurement is made at non-zero velocity.

of dynamic force quadratures. The amplitude dependence of the force quadratures gives a physically intuitive 'dynamic force curve' that can be measured without assuming a particular interaction model. Force quadrature curves are therefore extremely useful for gaining a deeper understanding of the tip–surface interaction.

2. Force measurement

Atomic force microscopy is rife with acronyms that distinguish its many modes of operation, some of which differ in subtle ways. All modes have one thing in common: they achieve image contrast by monitoring minute changes in the force between a surface and a sharp tip placed at the end of a flexible cantilever. Analytic AFM begins with an accurate and sensitive measurement of this force, where the cantilever beam acts as a linear transducer of tip-surface force.

The interaction force between the tip and surface $\vec{F}_{TS}(t)$ gives rise to tip motion $\vec{d}(t)$ which is recorded by monitoring the deformation of the cantilever beam (see Fig. 2). The force and deflection vectors have three components, but the standard AFM with a 4-quadrant photo detector can only resolve two signals, often referred to as the vertical and lateral deflection signals. The component of force normal to the plane of the sample surface F_z gives a vertical deflection of the tip d_z, causing a flexure of the beam. The component of force parallel to the surface and perpendicular to the major axis of the beam F_x gives a lateral deflection of the tip d_x which causes a torque and twist of the cantilever beam around its long axis. In the following we will drop the subscripts of force and deflection, as all discussion is valid for any component of the vectors.

Note however that the third component of force on the tip F_y also gives rise to a bending of the cantilever beam, which the standard detector can not distinguish from bending due to a normal force F_z. As long as the frictional or in-plane force F_y is not too large, the transverse bending moment will not be a big problem because the cantilever is typically rather stiff to such bending. However, when pushing against a surface, for example to calibrate the optical beam deflection system using the calibrated AFM z-scanner, significant frictional forces will give rise to errors that will be larger for stiffer cantilevers.

Quantitative AFM requires a well-calibrated measurement of at least one component of the tip-surface force. Calibration is required for 1) conversion from measured detector voltage to cantilever deflection and 2) conversion from deflection to force. Let us first consider the latter conversion where there are two basic types: Quasi-static, where we assume that the tip-surface and cantilever forces are balanced and the cantilever is effectively at rest, and dynamic, where we account for non-zero velocity and acceleration of the cantilever.

2.1. Quasi-static

The cantilever force is equal and opposite to the tip-surface force and the two are assumed to be in quasi-static equilibrium.

$$F_{\text{cant}} = -F_{\text{TS}} \tag{1}$$

The quasi-static assumption for the cantilever means that its motion is slow enough such that we may approximate the measured deflection as simply proportional to the cantilever force.

$$d = -\frac{1}{k_s}F_{\text{cant}} \tag{2}$$

Thus, calibration requires the determination of one static force constant k_s which tells us how much deflection we get in response to some tip-surface force.

The Hooke's law relation Eq. (2) is deceptively simple, but its application requires that we measure slow enough to ensure quasi-static equilibrium. The measurement time must be long in comparison to various time constants, for example the inverse of the cantilever resonant frequency, and the relaxation time given by the ratio of viscous to elastic force constants. The latter can vary widely depending on the material interacting with the tip and the medium through which the body of the cantilever moves.

The most important drawback of quasi-static force measurement is its inability to say anything about the viscous nature of the tip and surface materials. Viscous forces are, by definition, velocity dependent, and the quasi-static assumption is that velocity is negligible. The quasi-static method also has an important technical disadvantage in that it is inherently subject to excess low frequency noise or instrument drift (also call $1/f$ noise). Dynamic force measurement does not suffer from these drawbacks.