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Tip-sample interaction in tapping-mode scanning force microscopy

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Tip-sample interaction in intermittent contact scanning force microscopy, also called tapping mode, is experimentally studied to determine under which conditions tip-sample contact is established. Force vs distance curves are made while the cantilever is oscillating at its resonance frequency. Cantilevers with different force constants driven at different oscillation amplitudes have been used. In addition, samples with different hardness, such as silicon oxide, glass, and highly orientated pyrolytic graphite were taken as sample surface. From the analysis of the data we conclude that by choosing appropriate operating conditions, tip-sample contact can be avoided. This operating regime is of general interest in scanning force microscopy, since it allows imaging of even the softest samples.

INTRODUCTION

The physics and chemistry of surfaces is an appealing field since many processes such as chemical and biological reactions occur on then. Correspondingly, surface science is an important and well-established discipline. In particular, one topic that has drawn much attention is the interaction between surfaces. More recently, the scope of endeavor has broadened to include liquid structure, surface and thin-film phenomena. Not only are static forces being investigated but also dynamic (e.g., viscous and time-dependent) forces. For a detailed understanding of the corresponding processes, investigations on an atomic and molecular scale are needed. Techniques such as the surfaces force apparatus,[1] He-scattering, or scanning probe microscopy have been important tools for these kinds of investigations. Scanning force microscopy[2] (SFM) has become a powerful tool for studying surfaces on a nanometer scale. A variety of different operation modes have been developed to image not only the surface topography but also to probe its physical properties, such as magnetic[3] or electric[4] properties, but also liquid structures adsorbed on solid surfaces.[5] The typical way of using SFM is contact mode: that is, the tip is brought into mechanical contact with the sample and the topography of the surface is scanned. One problem of this method is that weakly adsorbed samples are easily damaged by the tip during the scanning process. Intermittent contact scanning force microscopy (IC SFM), also called ‘‘tapping mode,’’ [6] is one of the most extended modes, since lateral and shear forces are minimized thereby reducing damage to the surface. Therefore, this mode is specially suited for imaging soft and weakly attached materials such as biological samples[7] and polymers.[8,9] In addition to being a very useful technique in SFM, IC SFM can be applied for studying interaction between surfaces from a fundamental point of view. This is the topic of the present paper.

IC SFM essentially works as follows: the tip is oscillated at rather large vibration amplitude, that is, between 10 and 100 nm. As the tip is approached to the sample, the vibration is reduced due to tip-sample interaction. This reduction of vibration amplitude is used as feedback signal for the acquisition of topographic images. Additionally, the phase relation between the exciting signal and the response of the cantilever can be measured as a complementary signal, which seems to be related with the chemical composition of the surface,[10,11] or to the dissipation processes in the tip-sample system.[12,13]

Several theoretical approaches[14,15] have been proposed to interpret IC SFM mode images and to determine the contribution of the different forces and interactions involved in the acquisition process. Modeling as well as understanding of IC SFM is difficult mainly because of two reasons. First, different interactions may be relevant: van der Waals forces, forces due to elastic and even inelastic deformations, as well as adhesion forces between tip and sample.[1,16] Moreover also viscous forces due to the formation of a liquid meniscus or due to air damping may act and induce energy dissipation.[17] Second, the oscillation amplitude of the tip is much larger than the typical length scales related to the variation of tip-sample interaction. Therefore, modeling of IC SFM as a damped forced harmonic oscillator is not a valid approach. In fact, in the case of IC SFM the tip-sample system is a highly nonlinear problem and should be treated accordingly.[18,19]

Due to the complexity just described, we feel that right now there is no general agreement on exactly how IC SFM works. In fact, Whangbo, Bar, and Brandsch state that ‘‘to simulate any realistic experimental situation . . . leads to the problem of having more unknowns than equations.’’ [20] Nevertheless we think that the most accepted view is as follows. When the tip oscillates at a rather large distance, that is, when the lower turning point of the oscillation is of the order of a few nm from the sample, van der Waals forces might result in a small variation of the oscillation frequency and in a correspondingly very small variation of the oscillation amplitude. This effect is, however, generally believed to be quite small. As the tip further approaches the sample, the tip will touch the sample and feel the strong adhesive forces related to the tip-sample contact as well as to the formation of a liquid meniscus. This will induce a shift of the resonance frequency, and a high dissipation of energy. Both effects lead to a strong reduction of cantilever oscillation, which is used as a feedback signal. Finally, when the tip is further approached and strongly hits the solid surface, it will feel a strong repulsive force due to the elastic restoring force of the surface. This description can be restated in terms of the reduction of vibration amplitude: for very small reduction of vibration amplitude, van der Waals forces might be relevant, for larger reduction the main interaction should be due to adhesive forces, and for very strong reduction, the elasticity of the sample becomes relevant.

An important point in IC SFM is that, as the name indicates, it seems to be generally accepted that the tip touches the surface under usual operating conditions. In fact, most theoretical studies on IC SFM assume *a priori* that tip-sample contact occurs and accordingly, model the response of the system by some kind of repulsive interaction. Evidently the issues of tip-sample contact and of optimum imaging conditions in IC SFM are crucial. Moreover, the study of tip-sample interaction is an important point not only for the SPM community, but also for basic and applied research. However, in the vast literature, we have found only one early experimental study that proposes a direct way of observing this contact.[21] A detailed discussion of the method is given below. From that early study by Putman *et al.*,[21] the formation of a contact is inferred. In the present paper we have found in similar experiments that a careful analysis of the data leads to the result that for rather soft cantilevers and low vibration amplitudes, this is not correct. In these cases tip-sample contact does not occur. This behavior has been reported previously for rather special operation conditions,[22] as well as for dull tips.[23]

MEASUREMENT OF TIP-SAMPLE INTERACTION

The early work cited above[21] as well as the results presented here are based on the acquisition of force vs distance curves. This method is fundamental for a correct measurement of tip-sample interaction, since it allows the precise determination of the tip-sample distance during the experiment, and unambiguously to resolve the issue of contact formation. This is not directly possible in the kind of experiments that are now usually performed, where the amplitude and / or the phase of the oscillation amplitude is recorded as a function of piezo displacement.[24,25] In this context it is important to realize that the piezo displacement does not directly correspond to the tip-sample distance. To determine precisely the tip-sample distance, the simultaneous recording of cantilever deflection (normal force signal) is needed.

The experiments presented here were performed using a commercial SFM system.[26] The sample is fixed to a piezo tube that allows motion along the X, Y, and Z directions. The cantilever is glued to a small piezo plate to excite mechanical oscillations. To maximize the flexibility of the system the microscope is controlled by means of a digital signal processor inserted in a PC expansion slot.

The experiments described in this paper have been performed in jumping mode[27] while the tip is oscillated at its resonance frequency with different amplitudes. Prior to any experiment the sample surface is inspected using IC SFM. Then a clean and flat spot is selected and the X-Y scan is stopped. At this point the cantilever, being still in IC SFM, oscillates at the resonance frequency with a reduced amplitude that is previously selected. To analyze the precise dependence of the cantilever oscillation with tip-sample distance, jumping mode is used. In brief, jumping mode is a succession of force vs distance curves[28] with a feedback period between them at a given normal force set point. Jumping plus feedback times are of the order of a few milliseconds. Since the smallest cantilever resonance frequency is about 40 kHz, the tip oscillates more than 500 times during a single jumping cycle. Notice that while tip motion is induced by the small piezo where the cantilever is fixed, the jumping motion is performed by the sample piezo tube. The data have been recorded in the X-Y mode of an oscilloscope. The sample motion was input through the horizontal channel and the signal corresponding to the tip motion was input through the vertical channel. The oscilloscope image is then recorded using a digital video camera. In these images, the oscillation of the cantilever is not resolved due to its high frequency compared to the acquisition time of the whole force vs distance curve. These images can be interpreted as a time-averaged probability for finding the cantilever with a certain deflection for the different tip-sample distances. This explains why the borders of the curves are dark: at the turning points the time probability of a classical oscillator is maximum. We note that while the cantilever motion cannot be resolved in the oscilloscope images shown, this is not due to a limited bandwidth of our detection system, which is about 2 MHz.[29]

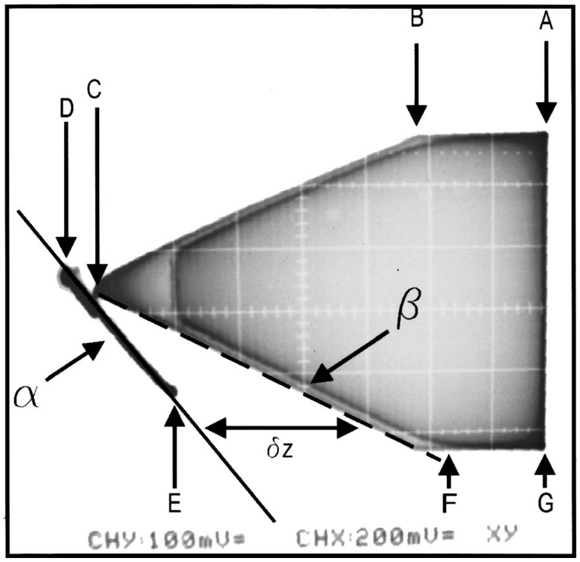

FIG. 1. Oscilloscope trace of the cantilever deflection vs z-piezo position. The relevant aspects of the experiment are shown as the tip goes through several states. Between A and B the oscillation is undisturbed and the amplitude is maximum. From B to C the amplitude decreases approximately linearly. At point C there is the snap to contact of the tip to the surface. As the z piezo further approaches the surface, the cantilever deflects upwards until the set point of the normal force is reached (point D). After a few milliseconds, the piezo is withdrawn. The tip separates from the sample at point E. Then the cantilever oscillates again, following the same linear regime as between B and C, until point F. Then the cantilever oscillates free again reaching finally the maximum tip-sample distance, G. Lines α and β are shown as guidelines: the solid one α defines the position of the surface, and β, dashed line, the lower turning point of the oscillation. The horizontal arrow marks the distance δz between the surface and the tip, at any given point.

To help the forthcoming discussion several labels have been included in Fig. 1 to mark the relevant points. We define point *A* as the starting point for an approach and retract cycle. At this point the sample is at the furthest position and the cantilever, free at this position, is oscillating with maximum amplitude. At point *B* some kind of interaction appears causing a reduction of oscillation amplitude. Usually, feedback is engaged in this regime to acquire images in IC SFM. This reduction is approximately linear with *Z* position. At point *C* the tip jumps onto the surface, then tip-sample contact occurs and the oscillation stops. As the sample is further approached to the sample, the contact force gets larger until the set-point is reached (point *D*). After a few milliseconds the *Z* motion is reversed and the tip is released at position *E*. At this point the cantilever resumes its oscillation, which increases linearly with sample motion until the free oscillation amplitude is reached (point *F*).

The lines α and β are drawn as guidelines and are fundamental for the correct evaluation of the experimental data. Line α (solid line) goes through points where the tip is in contact with the surface. This line, therefore, defines the surface position neglecting deformations of tip and sample. For any data point on the force vs distance curve, the corresponding tip-sample distance can be read off as the horizontal distance to this line α (see Fig. 1). In our case, since the deflection is measured in nm, it can also be read off as the vertical distance to that line. Line β (dashed line) is defined by the lower turning point of the cantilever oscillation.

The fundamental feature that is observed in many of our experiments is that as the vibration amplitude of the cantilever is reduced between points *B* and *C*, the tip does not touch the surface. In fact, the force vs distance curve, and more precisely line β does not reach the line α between points *B* and *C*. Figure 2 shows several of these force vs distance curves for different oscillation amplitudes and cantilevers with different force constants. While Figs. 2(a)–2(c) correspond to experiments acquired with a cantilever of about 50 N/m, Figs. 2(d)–2(f) correspond to a softer cantilever with a force constant of about 3 N/m. The cantilevers were driven at different oscillation amplitudes: about 10 nm [Figs. 2(a) and 2(d)], about 35 nm [Figs. 2(b) and 2(e)], and about 55 nm [Figs. 2(c) and 2(f)]. Note that in all the cases the tip does not touch the surface. Several different hardness substrates have been used for the experiments, such as highly orientated pyrolytic graphite (HOPG), glass, and silicon oxide.

In addition to the lack of contact in our curves, some other interesting observations can be made from our experiments. One of them is the relative slope of line β, compared with α. We normalize the slope of both lines such that the slope of line α is one.[30] The slope of line β is related to the length scale on which the interaction causing amplitude reduction is effective. For high slopes, that is, slopes near 1, this interaction increases very strongly with tip-sample distance, while for smaller slopes this increase is more gentle. We find experimentally that this slope is near 1 for soft cantilevers and as small as 0.35 for harder ones. We think however, that this dependence on the cantilever force constant is indirect: for hard cantilevers this slope decreases with time and with increasing adhesion [see Figs. 2(a), 2(b), and 2(c)].[16] Since adhesion is proportional to the radius of curvature of the tip,[31] we believe that due to the high loading force during tip-sample contact the tip becomes dull with time. For softer cantilevers, the same deflection of the cantilever induces a much smaller force. Thus the tip is not damaged, and correspondingly the curves are more stable [see Figs. 2(d)–2(f)]. Therefore we suggest that the slope β depends mainly on the tip radius rather than directly on the force constant.

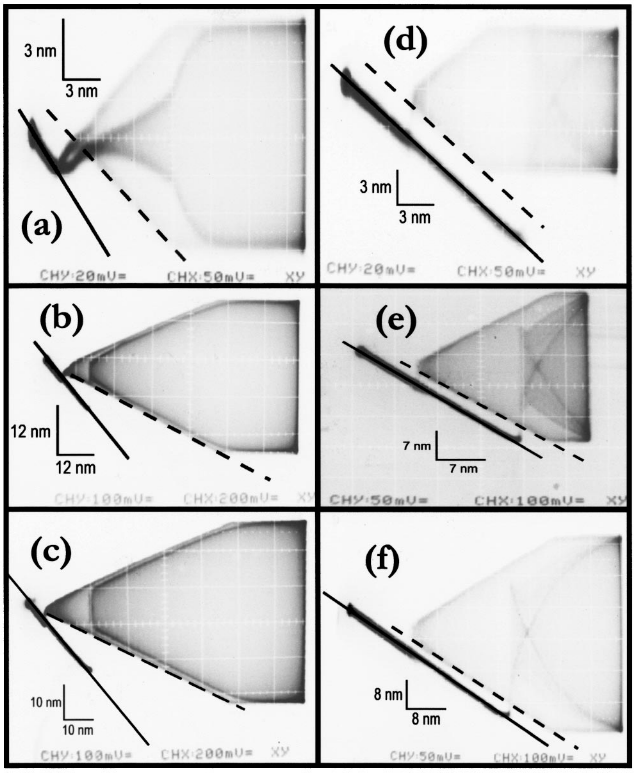

FIG. 2. Several deflection vs distance oscilloscope traces with different cantilevers and free amplitudes. In panels *a*, *b*, and *c* the force constant of the cantilever is about 50 N/m and its resonance frequency about 360 kHz. It is the same cantilever for the three measurements. In panels *d*, *e*, and *f* the force constant of the cantilever (the same always) is 3 N/m and the resonance frequency about 40 kHz. The free (maximum) amplitude is panels *a* and *d*: 10 nm, panels *b* and *e*: 35 nm, and panels *c* and *f* about 55 nm. The sample is: silicon oxide for panels *a*, *d*, *e*, *c* and *f*, and HOPG for panel *b*. The solid and dashed lines are guidelines with the same meaning than in Fig. 1. The order in taking the data are: panels *a*, *b* and *c* for harder cantilever (50 N/m), and panels *d*, *e*, and *f* for softer cantilever (3 N/m). Note that adhesion force of tip to surface grows with time.

Another interesting feature in our data is that, if the oscillation amplitude $a_{\rm osc}$ is low enough, the maximum restoring force $F_{\rm osc}$ of the cantilever during oscillation is lower than the adhesion force $F_{\rm ad}$ which is measured during the receding part of the force vs distance curves, i.e., $F_{\rm osc}=ca_{\rm osc}<F_{\rm ad}$ [see Fig. 2(d)]. This observation is not consistent with the formation of a contact between tip and sample, since the tip would then stick to the surface and the oscillation would stop. Therefore we again conclude that tip-sample contact does not occur in this case.

To avoid confusion, and to make clear the point that the tip can indeed touch the surface, in Fig. 3 we show a curve where the tip-sample contact does indeed occur, as one would expect for this so-called ''tapping mode.'' The difference between the data shown here and the data shown in the previous figures is essentially the oscillation amplitude of the

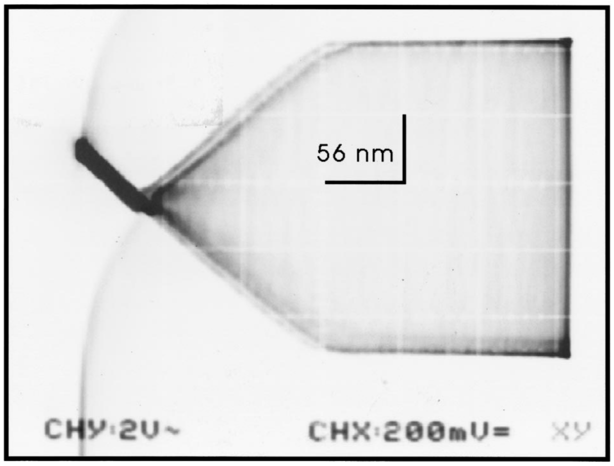

FIG. 3. Deflection vs distance oscilloscope for a soft cantilever (0.75 N/m) with a large free oscillation amplitude. As described in the main text, in this case the tip does touch the surface due to the high oscillation energy in the cantilever.

cantilever. We have found that for large vibration amplitudes and hard cantilevers the tip rather touches the surface,[32] while for soft cantilevers (about 1 N/m) and low oscillation amplitude (10–50 nm) the tip rather stays oscillating in the noncontact regime. We note, however, that for very soft cantilevers and very low oscillation amplitude, the tip might jump directly into mechanical contact with the sample without showing any reduction in oscillation amplitude, that is, the ''tapping'' regime between the points *B* and *C* in Fig. 1 is absent. We think that a very small as well as a very large radius of curvature of the tip also tends to keep the tip-sample system in the noncontact regime.

CONTACT VERSUS NONCONTACT

The experimental conditions under which the noncontact regime is observed can be expressed more conveniently in terms of oscillation energy, rather than the two parameters force constant and oscillation amplitude. From what has been discussed above, it follows that for low oscillation energy the tip does not touch the sample, and for high oscillation energy, it does. This can be understood rather easily as follows: Assuming, as we know from other experiments, that the main interaction in this regime is of viscous type, then the reduction in oscillation amplitude can be related to the energy dissipated in the tip-sample system:

$$\Delta E_{\mathrm{diss}}(z)=E_{\mathrm{free}}-E=\frac{ca_{\mathrm{free}}^2}{2}\left[1-\left(\frac{a(z)}{a_{\mathrm{free}}}\right)^2\right],$$

where c is the force constant of the cantilever, E_{free} and a_{free} are the free oscillation energy and amplitude, and $E(z)$ and $a(z)$ are, correspondingly, the oscillation energy and amplitude at a given point z of piezo Z displacement. Note that tip-sample interaction is very small in the noncontact regime as compared to the case where the tip touches the sample and that the energy that is dissipated is not measured directly but through the reduction factor $r(z)=a(z)/a_{\mathrm{free}}$. Therefore, the smaller the total energy in the cantilever oscillation, the better the resolution for energies is. To see this directly, the equation above can be easily rewritten as

$$\Delta E_{\mathrm{diss}}(z)=E_{\mathrm{free}}[1-r(z)^2].$$

Compared with experiments performed typically in ''tapping mode'' ($c\approx 50$ N/m, $a_{\mathrm{free}}\approx 50$ nm, and sometimes even more than 100 nm) for the experiments discussed here where a noncontact regime is observed ($c\approx 1$ N/m, $a_{\mathrm{free}}\approx 10-50$ nm) the energy stored in the oscillation of the cantilever is between two and five orders of magnitudes lower. This correspondingly means 100 to 10 000 times more sensitivity to small interactions. We, therefore, believe that although this noncontact very probably also exists for large oscillation energy, in this case it is simply not observed.

A fundamental question in the interpretation of our data concerns the mechanism for the interaction observed in our experiments. Unfortunately, we still do not understand its precise nature, but it does not seem to depend strongly on the chemistry of the sample. Moreover, we have found that this interaction is of a dissipative kind,[29] that is, it is not induced by a conservative but some kind of viscous force. Several mechanisms such as liquid necks,[33] vapor condensation, air friction, or a higher viscous force which acts on the tip due to an increased vapor pressure just above the surface, might be responsible for this interaction. The precise mechanism underlying this interaction is still under investigation.

IMAGING APPLICATIONS

Finally we would like to discuss imaging applications, since we believe that this will be the most important use of this noncontact dynamic SFM. In fact, with low oscillation energy (about 4×10^{16} J) and reduction factors $a_{\mathrm{set}}/a_{\mathrm{free}}$ of 0.95–0.4 we obtain the best images with high resolution on rather soft and/or weakly adsorbed samples such as carbon nanotubes and DNA strands,[34] as well as water layers.[35] Incidentally, if these samples are acquired in jumping mode, where we know for sure that a mechanical contact is established, we observe that the samples are often moved by the tip even though lateral forces due to the scan motion are not present, and both normal force and contact time are known. We never observe this in IC SFM under the conditions described above, and believe that this is yet another proof for the absence of tip-sample contact.

In addition we should comment that from IC SFM images of carbon nanotubes on SiO_2 and DNA strands over mica, we deduce tip radii as small as 15 nm. Amplitude reduction experiments carried out with the same cantilever, oscillation amplitude, and sample before and after the image, did not show any trace of contact. Thus, the lack of contact cannot be attributed, at least in this case, to a dull tip.

SUMMARY

The amplitude reduction of an oscillating cantilever has been studied as a function of tip-sample distance by acquiring force vs distance curves while the cantilever is oscillating at its resonance frequency in ambient pressure. We suggest that these kinds of experimental curves should be generally used to determine whether tip-sample contact occurs, and to measure tip-sample distance with accuracy. From our data we conclude that the reduction in oscillation amplitude cannot be due to a direct tip-sample contact if the total oscillation energy in the system is small. Tip-sample contact has been described as the main issue responsible for the amplitude reduction of the cantilever oscillation.[36] This is why this SFM mode is sometimes referred to as ‘‘tapping mode.’’ From the experiments presented here, we propose that this terminology should be reconsidered, since in quite a few cases tip-sample contact does not occur. We propose the general terminology dynamic SFM, and a differentiation between contact and noncontact dynamic SFM. We believe that dynamic noncontact SFM in ambient conditions is of great value for the study samples such as DNA or carbon nanotubes, or even liquid structures adsorbed on solid surfaces.

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[28] With force we mean in the present context cantilever deflection.

[29] We use a special chip, AD 880 by Analog Devices that has a maximum bandwidth of 40 MHz, which we limit to about 2 MHz to reduce noise at high frequency.

[30] For the points on line α, tip and sample are in contact, therefore they move together. That is tip movement is equal to sample movement.

[31] The adhesion force is $F_{\mathrm{adh}}=4\pi\gamma R_{\mathrm{eff}}\cos\varphi$, where φ is the contact angle of the meniscus around tip and sample, γ is the surface energy of water, and R_{eff} is an effective radius of curvature of the system, such that $1/R_{\mathrm{eff}}=1/R_{\mathrm{surf}}+1/R_{\mathrm{tip}}$ with R_{tip} tip radius and R_{surf} local radius of curvature of the sample; see also Ref. 1.

[32] This is correct as long as the tip is not severely damaged, otherwise again the tip might not touch the sample (Ref. 23).

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