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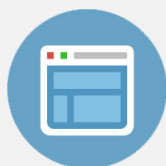
Measurement of the absolute separation for atomic force microscopy measurements in the presence of adsorbed polymer

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We demonstrate that the absolute separation between an atomic force microscope (AFM) tip and a solid substrate can be measured in the presence of an irreversibly adsorbed polymer film. The separation is obtained from the analysis of a scattered evanescent wave that is generated at the surface of the solid. By comparing our scattering measurements to conventional AFM measurements, we also show an example where a conventional AFM measurement gives the incorrect force-distance profile. We validate the measurement of separation from scattering by examining the force-separation profile in the presence of surfactant solution. This validation is possible because the tip can be used to desorb the surfactant film that forms in surfactant solution, so we are able to measure both the scattering and the cantilever deflection when the tip is in contact with the solid substrate. The main limitation of our technique now is the lack of a rigorous method for predicting the intensity of scattering from the tip in contact with a solid that is coated with a film. © *2006 American Institute of Physics.* [DOI: 10.1063/1.2202929]

INTRODUCTION

An atomic force microscope (AFM) is widely used to determine the forces between polymer-coated solids.[1–4] A fundamental limitation of an AFM surface force measurement is that the separation between the solids is not measured explicitly; the separation is inferred from the force measurement. In conventional AFM measurements, the relative separation is obtained by adding the deflection of the cantilever to the displacement of a piezoelectric translation stage[5] ("piezo displacement"). The absolute separation is determined by the interpretation of the force-distance profile. Usually, one defines zero separation to be where there is a very steep gradient in the surface force. This procedure is acceptable when the solids are incompressible and the adsorbed material can be displaced at an accessible force. However, when the solids or attached films are compressible, problems arise in determining the cantilever deflection contribution to both the relative and absolute separation.

The relative separation can be obtained through a calibration step. For example, when measuring the forces between rigid solids coated with a physisorbed polymer layer, one usually measures the force between the solids prior to adsorption. In this step, the cantilever deflection slope (deflection versus piezo displacement) can be calibrated by pushing against the rigid solid, so the relative separation and force can be measured in the presence of the adsorbed polymer. The problem arises when determining the absolute separation in the presence of the polymer. The cantilever often cannot generate enough force to displace the polymer, so the

tip cannot reach zero separation. There may also be a very steep gradient of force when there is a significant thickness of the polymer film between the solids, thereby confusing the signature of zero separation.

A low cantilever spring constant is desirable for high resolution of weak forces in thick films, but a weak spring makes it difficult to distinguish between the stiffness of the solid and the composite stiffness of the solid plus polymer film. In practice one must compare the deflection slope of the spring during the calibration against a hard surface to the very similar deflection slope during compression of the polymer. This comparison must be made over the large range of spring movement that is required to displace the polymer film. Also the stiffness of the polymer layer usually increases as the film is compressed, which results in a deflection slope that asymptotes to a value that cannot be resolved by a force gradient measurement.

In principle the absolute separation in the presence of a polymer film could be determined simply by measuring both the piezo displacement and deflection of the spring in the range of zero separation (direct contact between bare solids), and then remeasuring the piezo displacement and the deflection of the spring at all subsequent times. Sadly, this procedure is always defeated by small changes in the dimensions of the AFM caused by thermal drift.

A much better solution is to explicitly measure the separation between the solids. In the surface forces apparatus,[6] the separation is measured explicitly by interferometry. In recent work[7–9] we have shown that the scattering of an evanescent wave can be used to measure the absolute separation between a tip and a sample or a colloidal probe and a sample. The scattering from a silicon nitride AFM tip varies expo-

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nentially with separation, so it is simple to obtain the separation from scattering. For a colloidal probe, the scattering is nonexponential so a calibration curve or a more complex theory is required.[7]

Here we demonstrate the usefulness of evanescent wave atomic force microscopy[8] (EW-AFM) for measuring forces in polymer solutions. We focus on experiments with silicon nitride AFM tips because we have already established that the scattering is proportional to the intensity of the undisturbed evanescent wave, and therefore we can account quantitatively for the refractive index of the solution between the tip and the sample.[9] The intensity of an evanescent wave in the absence of a tip is given by

$$I_z^E = I_0^E \exp\left(\frac{-z}{\delta}\right), \tag{1}$$

$$\delta = \frac{\lambda}{4\pi\eta_2\beta}, \tag{2}$$

$$\beta = \sqrt{(\eta_1/\eta_2)^2 \sin\theta_I - 1}, \tag{3}$$

where I_0^E and I_z^E are the intensities of the wave at zero distance and at a distance z, from the surface of the solid. The decay length of the wave δ depends on the wavelength of light λ, the refractive index of the incident and transmission medium η_1 and η_2, and the incident angle θ_I. By experiment, we find that the scattering intensity from a silicon nitride tip measured below the sample I_h is proportional to I_z^E.

$$I_h = I_0 \exp\left(\frac{-h}{\delta}\right). \tag{4}$$

Here h represents the separation between the tip and the sample, and δ is the same as Eq. (2). Clearly, the separation h can be obtained from Eq. (4) by measuring I_h and I_0.

MATERIALS AND METHOD

Experimental method

The evanescent wave atomic force microscope design has been described in a previous publication.[8] It is based on an Asylum Research MFP-3D AFM (Santa Barbara, CA), which is mounted on a Nikon TE2000 inverted light microscope (Coherent Scientific, Australia). The AFM measures the force acting on the tip, translates the tip normal to the interface, and measures the translation with a linear variable differential transducer (LVDT).

We used either a 405 nm (405-50-COL-004, Oxiuss, France) or a 532 nm diode laser (TECGL-10, World Star Tech, Toronto, Canada) to create an evanescent wave at the interface between a borosilicate glass plate (12-544-12, Fischer Scientific) and an aqueous solution. Water and surfactant results were obtained using the 532 nm laser. The polymer results were obtained using the 405 nm laser. The glass plate was coupled to a trapezoidal glass prism (Knight Optical, U.K.) using index-matching fluid (Nye Lubricants, Fairhaven, MA). The refractive index of the water was 1.333, and the refractive index of the glass plate was 1.515. For the 532 nm laser the incident angle was approximately 75°, so the decay length was 70 nm. For the 405 nm laser the

corresponding values were 76° and 52 nm. The scattering signal produced by AFM tips interacting with the evanescent wave was collected by a Nikon LU Plan 50×/0.55NA objective and transferred to a photomultiplier tube (H5784-20, Hamamatsu).

Fluid exchange during experiments was achieved by altering the Asylum cantilever mount to hold two lengths of PEEK tubing (Valco Instruments, Houston, Texas) in close proximity to the cantilever. This enabled us to exchange fluid in a capillary of liquid formed between the cantilever mount and the surface.

Experimental materials

The AFM tips used in these experiments are silicon nitride square pyramid cantilevers (NP, Veeco, Santa Barbara, CA), which were cleaned under ultraviolet light prior to use. These cantilevers are manufactured with a square pyramid tip that is hollow and 4 μm high, with an effective tip radius of less than 60 nm. They have 15 nm of chromium and then 60 nm of gold deposited on the side facing away from the sample. All solutions were prepared in water purified in an Easypure UV system (Barnstead, Dubuque, IA); the water had a resistivity of 18.2 MΩ cm and a surface tension of 72.3 mN/m. The borosilicate glass slides (12-544-12, Fischer Scientific,) have a rms roughness of 0.26 nm over an area of 5 μm^2. The glass slides were used immediately after they were cleaned by O_2 plasma for 45 s at 250 mTorr, using a Plasma Prep II (SPI Supplies, West Chester, PA).

Dr. Greg Qiao and Dr. Tiziana Russo, of the University of Melbourne, kindly provided the zwitterionic polymer used in these experiments. It is a polysulfobetaine[10–12] synthesized by the polymerization of N,N-dimethyl-N-[2-[(2-methyl-1-oxo-2-propenyl)oxy]ethyl]-3-sulfoethanaminium. These polymers are insoluble in water; all polymer studies were performed in solutions containing 100 mM NaCl. The sodium chloride (Aldrich 99.99%) was roasted for 6 h at 560 °C. Didodecyldimethylammonium bromide (DDAB) (Fluka, ⩾98%) was used as supplied.

RESULTS

The results section is organized in a progression of experiments that are of increasing difficulty to analyze. First we describe the measurements when there is only salt solution between the tip and sample, then we describe the measurements when there is a thin organic film than can easily be displaced by the AFM tip. Finally we describe the case when the film cannot be displaced.

Figure 1 shows the concordance between conventional AFM analysis of separation and the separation measured from evanescent wave scattering [Eqs. (2)–(4)], when there is not a thin organic film between the solids. The force-separation profile measured between the hydrophilic tip (0.12 N/m) and hydrophilic glass surface, in 100 mM NaCl, is purely repulsive on approach and withdrawal, and for clarity, we have only plotted the approach data.

Concordance between conventional AFM separation measurements and scattering measurements in the presence of a thin film of low molecular weight surfactant is shown in
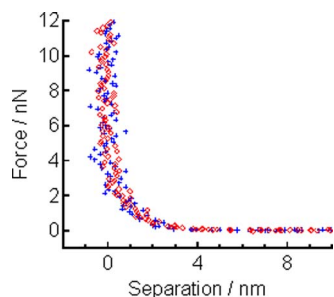
FIG. 1. The force-separation profile measured between a hydrophilic Si_3N_4 tip ($k=0.12$ N/m) and a hydrophilic glass surface in 100 mM NaCl. The separation between the surfaces was determined by conventional AFM techniques (blue crosses), and by using Eq. (2) to determine the separation from the scattering of the AFM tip (red circles). Scattering and deflection data were collected simultaneously.

Fig. 2. In this case the surfactant film is easily displaced by the AFM tip. The thin film was prepared by adsorption of didodecyldimethylammonium bromide in equilibrium with a 0.5 mM solution in bulk. The scattering data in this case are more complex because of the more complex force profile. Figure 2(a) shows the raw data of the scattering intensity versus piezo displacement. In region 1, the force is negligible so the scattering is an exponential function of piezo displacement. As the separation between the two surfaces decreases, an electrostatic repulsion between adsorbed bilayers causes the AFM cantilever to deflect. This results in an *apparent* deviation from exponential scattering in the raw signal (region 2). When surfactant bilayers from opposing surfaces
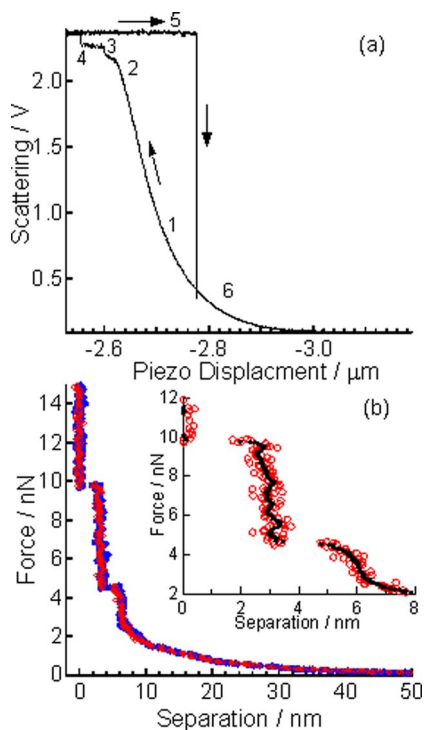

FIG. 2. Profiles measured for a Si_3N_4 tip ($k=0.12$ N/m) and a glass solid in 0.5 mM DDAB. (a) Scattering intensity as a function of piezo displacement. The profile shows hysteresis due to the adhesion of the tip to the glass. (b) Force as a function of separation measured by conventional AFM analysis (blue circles) and EW-AFM (red circles). The inset shows scattering data only on an expanded scale. The black line is running average of 10 points.

begin to interact, the rate of approach of the AFM tip to the glass surface decreases, and the scattered intensity levels off. When a surfactant layer is pushed out from between the tip and surface a step in the raw scattering data is observed (region 3). After expulsion of the final surfactant layer (region 4), the tip and glass surface make hard contact and the scattering intensity is constant. As the piezo begins to retract, the tip and surface remain in contact until the force of the spring exceeds the force of adhesion (region 5). The surfaces then separate suddenly, and the scattering intensity jumps to the value measured on approach (region 6).

Comparison of the force-separation profile determined by the scattering intensity and by conventional AFM analysis for the approach only is shown in Fig. 2(b). Both methods produce the same force-separation profile. The separation for the scattering experiments was calculated using Eq. (2) and a refractive index for the transmission medium, η_2, of 1.333. The two force-separation profiles are concordant despite the fact that we ignored the refractive index contrast of the adsorbed surfactant film.

The inset in Fig. 2(b) shows detail of the region of surfactant exclusion as measured by scattering separation only (red circles). This figure can be used to demonstrate the resolution of the evanescent wave AFM. The surfactant is displaced from the thin film in two discrete steps from 5 to 3 nm, and from $\sim$2.5 to 0 nm. The data were collected at 0.44 Hz at 2000 points per second. If we take a running average of 10 points in the data, so that the acquisition rate is effectively 200 points per second (black line), we can clearly resolve the slope of the force-separation profile for the $\sim$3 nm film. The stiffness of the film is 3.2 N/m. The standard deviation of the averaged points from a straight line is 0.13 nm.

The force-separation profile shows the layering of the surfactant in the thin film between the tip and the sample. The all *trans* (maximum length) conformation of DDAB has a length of about 1.9 nm, but neutron-reflectivity measurements[13] are consistent with a thinner layer of about 1.1 nm. Our data show that the force gradient increases more rapidly starting at about 10 nm, which is consistent with the expected electrostatic double-layer force generated between charges solids in aqueous solution. There is a steep rise in force at $\sim$6 nm corresponding to a bilayer on each solid ($\sim$1.5 nm per surfactant layer). When these bilayers are compressed to about 5 nm they yield, and a new structure is formed with a thickness of 3 nm ($\sim$1.5 nm per surfactant layer). This very stiff layer is then compressed to about 2.7 nm before it yields and the surfactant is displaced from between the tip and the sample.

Polymer solution

Often an AFM cantilever cannot apply enough force to displace an adsorbed polymer from between the tip and sample. In this case it is necessary to have the correct value of I_0 prior to the adsorption of polymer in order to measure the absolute separation by our scattering method. We have tried two different methods (*reference* and *contact*) to obtain a value of I_0 prior to polymer film formation. The reference method involves measuring the scattering value of I_0 in the
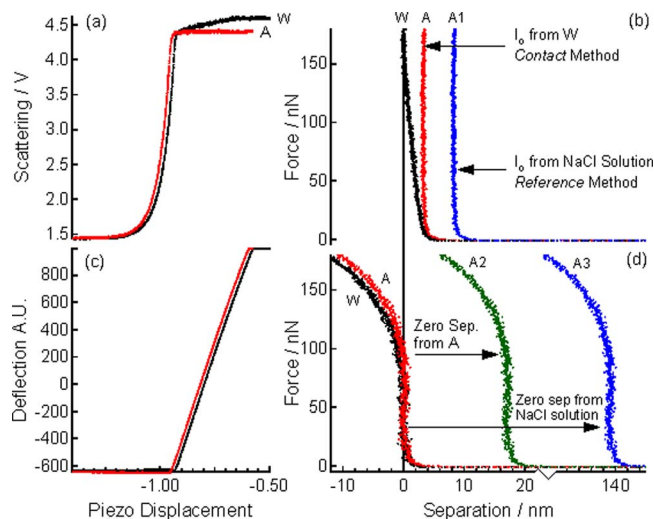
FIG. 3. (a) Raw scattering intensity. The intensity is greater at the start of the withdrawal because the tip is in contact with glass on withdrawal. (b) Force-separation profile measured using Eq. (4) for the separation. The red curve (A) is the film thickness using the *contact* method to determine the tip position on approach. The blue curve (A1) would be the separation if the *reference* method is used to determine separation. (c) AFM tip deflection captured simultaneously to scattering data in (a). (d) The force-separation profile determined using the AFM deflection data. The withdrawal (W) and approach (A) are coincident using conventional AFM analysis. The green curve (A2) would be the apparent film thickness if we assumed the displacement between approach and withdrawal runs is due to the thickness of the film. The blue curve (A3) would be film thickness if the piezo position prior to the addition of polymer was used to determine the tip position on approach.

solvent prior to the addition of polymer. This value of I_0 is used to determine the separation between the two surfaces after polymer solution is added to solution and adsorbed to the solid. This method relies on the assumption that the adsorbed film does not change the magnitude of the tip scattering when the tip is in contact with the sample. This assumption was found to be valid for the surfactant experiment described above, but may not always be valid. The contact method involves adding the polymer solution while the tip remains in contact with the surface. I_0 is measured immediately before the tip is withdrawn from the surface and is then used to determine the separation between surfaces with adsorbed polymer. Both methods require a constant value of I_0. I_0 can in practice change due to the appearance of other scattering particles in the field of view, or a change in the intensity or position of the evanescent zone. To reduce the error associated with the latter, we altered the Asylum AFM cantilever mount to hold two tubes in close proximity to the cantilever. This helped us to exchange fluids between the tip and surface, without disturbing the position of the AFM tip in the viewing field of the photomultiplier tube (PMT), or in the ellipse of total internal reflection.

The results for both the reference and contact procedures are shown in Fig. 3. Figure 3(a) shows the scattering from a Si_3N_4 tip ($k=0.58$ N/m) in contact with a glass surface while a 1 mg/ml polymer-salt solution was added. After an adsorption time of 5 min, the tip was withdrawn (W) and then brought back to the surface (A). The piezo was run at a rate of 200 nm/s over a scan range of 4.2 μm; with a 10 s dwell away from the surface. While the tip was away from

the surface ($\sim$1 min), polymer adsorption occurred in the space previously occupied by the tip. When the tip was brought back towards the surface (A), the newly formed polymer layer kept the tip from contacting the glass surface. This results in a measured tip scattering intensity, at the point of closest approach, that is less than the scattering intensity measured when the tip was in direct contact with the glass. Clearly the tip is not in contact with the glass at the point of closest approach (A). Figure 3(b) is the force-separation profile obtained by using the scattering intensity to measure separation. When we use the maximum intensity measured before withdrawal to determine the separation (contact method), the surfaces begin to interact on approach (A) at a separation of $\sim$7 nm, and the film cannot be compressed to less than $\sim$3 nm. The contact method ensures that the maximum measured intensity during withdrawal (W) corresponds to zero separation. The reason for the hysteresis between the approach and withdrawal curves is that the high molecular mass polymer is not at equilibrium; it is not displaced during the approach cycle.

If we use the reference method to determine the separation between these surfaces, we get a different answer for both the onset of interaction and minimum film thickness ($\sim$12 and 8 nm, respectively, A1). The reference method also shifts the withdrawal curve (not shown) $\sim$5 nm, off the surface.

We observe both small increases and decreases in I_0 during the adsorption of the polymer film. These small changes make it difficult to use I_0 measured before the addition of polymer (reference method) to determine separation. Because these changes in intensity were random, we believe that they may have occurred because of the movement of the evanescent zone relative to the AFM tip. This problem may in future be corrected with a more rigid optical setup, and smaller gradients in evanescent intensity across the solid. For the remainder of this article we will use only the contact method because only this method produces consistent results that are physically reasonable.

Figure 3(c) is the deflection of the cantilever measured during the same experiment as (a). If we use conventional AFM analysis to analyze these data, we define the region with the steep and constant slope (region of constant compliance) to be the zero of separation. We cannot resolve a difference in slope between the constant compliance regions on approach and withdrawal, so we assign them both to zero separation [Fig. 3(d)]. Alternatively, we might note that the approach and withdrawal curves are displaced by 18 nm in Fig. 3(c), and assume that the film is 18 nm thick (A2). Clearly this is an error: the actual displacement varies between 3 and 7 nm. The discrepancy is due to unmeasured changes in the dimensions of the AFM, which vary depending on experimental conditions and time during the measurement.

We could also analyze the deflection data by a reference method. In this case we use the deflection and piezo displacement in contact before the addition of polymer as a reference for the measurements after the polymer was added. In this case we measure a thickness of 140 nm [Fig. 3(d), A3]. Clearly the drift in the AFM dimensions (140 nm) is
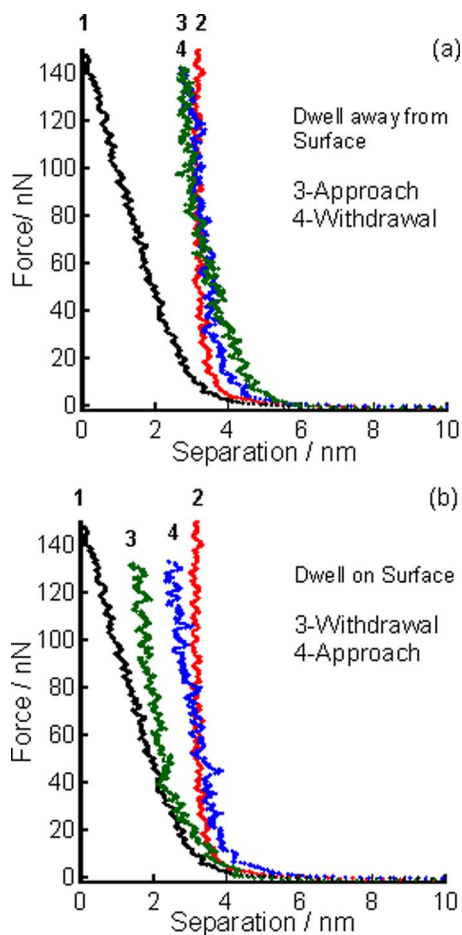
FIG. 4. Force-separation profiles determined using Eq. (4) in polymer-salt solution. The first withdrawal (1) and approach (2) are re-plotted from Fig. 3(a) approach-withdrawal collected after a 10 min equilibration time with the tip away from the surface. (b) The tip was left in contact with the polymer surface for 1 min, withdrawn (3), and then immediately brought back into contact (4). All data shown are the average of 10 measured points.

much worse than the "drift" introduced from I_0 reference method ($\sim$5 nm), making the deflection reference method totally impractical.

All three methods that one can use to determine the separation in the conventional AFM method give an *incorrect* offset to the force-separation curves. Note that the traditional analysis also gives the incorrect shape to the withdrawal force curve.

The scattering method has an additional advantage over the deflection method for determining zero separation. Note that at forces >100 nN, the deflection data indicate that the sample stiffness is decreasing at an accelerating rate. This unphysical situation is usually blamed on the nonlinearity of the diode response, and this region of data is discarded. In contrast, reliable separations can be measured by the scattering method in this force range.

The ability to determine zero separation enables EW-AFM to perform new measurements. To demonstrate this we have measured the time response of the polymer to applied loads. Figure 4 shows how the adsorbed polymer film relaxes after compression. Curves 1 and 2 show the force on withdrawal and approach, replotted from Fig. 3. After the primary withdrawal-approach curve, we left the tip away from

the surface for 10 min and then ran an approach (3)-withdrawal (4) curve [Fig. 4(a)]. The separation was determined using the maximum I_0 measured in curve 1. In Fig. 4(b) we left the tip in contact with the polymer film for 1 min, then withdrew the tip (3) and brought it back to the surface (4). These data show that (a) 10 min is enough time for the film to establish equilibrium thickness, and (b) the film thins with time under load. The latter was also observed by monitoring the scattering with time. Neither of these measurements are possible with conventional AFM analysis.

DISCUSSION

Effect of a thin organic film on the scattering profile

The deflection-scattering-displacement measurements in surfactant solution (Fig. 2) provide an interesting opportunity to investigate the effect of a thin film on the scattering from an AFM tip. The solid surface is very stiff, so we can obtain a reliable measurement of cantilever deflection. The stiff solid and the ability to displace the surfactant at high loads allow us to unambiguously obtain the absolute separation from a conventional AFM analysis. Thus we can obtain a plot of scattering as a function of film thickness. We find that the scattering profile agrees very well with the intensity profile of an evanescent wave in the absence of a thin film. This is shown clearly by the concordance between the two data sets in Fig. 2(b).

Thus, we have shown that we can measure the separation (by scattering) in the presence of a very thin film, even without knowing the thickness or refractive index of the film (or indeed without knowing whether the film is present at all).

Can we ignore the presence of films of all types? We should note here that the surfactant film is special in so far as surfactant molecules tend to pack into certain preferred geometries, and DDAB, in particular, packs into flat bimolecular sheets. Thus we would not expect a (broad) capillary of surfactant to form; instead we expect the film to remain as a flat sheet. The important point is that when a sharp tip contacts the surface, we expect that only a small fraction of the AFM tip will penetrate into the film.

Here we will briefly consider the effect of a thin film on scattering by an AFM tip. A detailed analysis of the scattering of the tip is beyond the scope of this article, we will simply discuss the effect of films on the intensity of the evanescent wave in the absence of the tip, and assume that the scattering that we measure is proportional to the intensity of the evanescent wave. This is in keeping with the success of the same model in the absence of the thin film.

When an evanescent wave passes through a series of slabs of different refractive index, the decay in each slab is given by Eqs. (2) and (3), using the refractive index for the particular slab. Thus, for a single film, the intensity of the evanescent wave at distances greater than the film thickness is given by

$$I_h = I_0 \exp\left(\frac{-t}{\delta_{\text{film}}}\right) \exp\left[\frac{-(h-t)}{\delta_{\text{bulk}}}\right], \tag{5}$$

where t is the thickness of the film, δ_{film} is the decay length in the film, and δ_{bulk} is the decay length in bulk solution. The

only h dependence is in the last term, so the scattering decay length at tip separations greater than the film thickness is given by the properties of the bulk. This explains why relative displacements are accurate in the presence of the film: the presence of the film changes the evanescence wave intensity outside the film, but not the decay outside the film.

The situation is more complex when the end of the tip is partially immersed in the film. As the tip approaches the solid, some parts of the tip displace the film, and some parts displace the bulk liquid. The exact dimensions of these sections are unknown but definitely smaller than both the decay length and the wavelength of light. The fact that the separation derived from the scattering data analyzed with the bulk refractive index gives the same separation as the conventional AFM analysis suggests that only a small amount of the scattering portion of the tip is immersed in the film. This is reasonable for a sharp tip and an evanescent decay length of 70 nm with a film thickness of only 6 nm: most of the scattering regions of the tip stay in the bulk liquid environment. It is reasonable to assume that the intensity of light that reaches sections of the tip outside the film is affected by the presence of the film [Eq. (5)]. In the contact method, we have measured I_0 in the presence of the film so this effect is rolled into the value of I_0. Thus, sections of the tip that never enter the film produce scattering that is consistent with Eq. (4). In the reference method, the addition of a film after the reference is measured will affect the scattering intensity from all parts of the tip.

In general, if we wish to have a scattering signal that is independent of film properties (e.g., when the film properties are unknown and unwanted), we should aim to have a long evanescent decay length (long wavelength, small incidence angle) and a sharp probe so that much of the scattering tip moves only through the "bulk" solution above the film. A long decay length also helps the reference method. Clearly, if we want to sense the refractive index of the adsorbed film, we need to have a blunter probe and a shorter decay length.

In conclusion we have demonstrated a new method to measure the separation between an AFM tip and surface. By measuring the scattering of an evanescent wave by an AFM tip in contact with a solid before the adsorption of a film, it is possible to measure the thickness of the film and also a complete force-separation profile without again contacting the tip to the sample. This allows us to accurately measure force-separation profiles in the presence of strongly adsorbing polymers.

ACKNOWLEDGMENTS

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