Direct measurement of the absolute value of the interaction force between the fiber probe and the sample in a scanning near-field optical microscope

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(Received 1 April 2002; accepted for publication 18 June 2002)

The absolute values of the force exerted by the fiber probe of a scanning near-field optical microscope onto the surface were measured using an atomic force microscope in ambient conditions. We demonstrate that a usually neglected static attraction force is dominant at small dither amplitudes and is of the order of 200 nN. The tapping component of the force, often referred to as shear force, is of the order of 1 nN at these conditions for both the tuning fork-based and optical in resonance detection schemes. Other peculiarities of the shear force interaction are also discussed. [DOI: 10.1063/1.1499736]

When closely approaching the surface with the laterally dithered fiber tip of a scanning near-field optical microscope (SNOM), a decrease in the dither amplitude is observed. This effect, which can be monitored optically or using a quartz tuning fork sensor, is routinely used to control the distance between the fiber tip of the microscope and the surface of the sample and is often referred to as shear force control (see, for instance, Ref. 1 for a recent review of the field). Starting from pioneering works,[2] dissipative van der Waals and capillary forces were proposed as possible explanations for the origin of shear forces. Later on, Gregor *et al.* invoked the so-called nonlinear bending force model with tapping contact caused by a small tilt of the fiber relative to the normal to the surface.[3] This model neglects energy dissipation and considers transfer of the energy of the vibrating fiber to the sample due to elastic collision. For dither amplitudes of 0.1–1 nm (attainable for tuning fork-based detection) forces ranging between 50 and 500 pN were calculated[4,5] while for dither amplitudes of the order of 10 nm (optical detection) a value of a few nN was reported.[6]

Despite all the popularity of the shear force-based SNOM the absolute values of the shear force have never really been *measured*, so researchers are obliged to rely on model-dependent estimations. Presented in this letter are measurements of the shear force performed by means of a reasonably stiff (to avoid the jump to or out of contact problems[7]) atomic force microscope (AFM) cantilever. The experiments were performed for the most popular shear force detection methods currently in use: resonance optical detection,[2] tuning fork-based detection[4] and out of resonance[8,9] optical detection (the latter results together with further discussion, will be published elsewhere).

All experiments were performed in typical indoor winter conditions: temperature of 18–20 °C and relative air humidity of 15%–25%. The experimental setup combines a home-made SNOM with a home built AFM. Both devices were used earlier to study nanolocal fluorescence resonance energy transfer in the geometry of the SNOM and AFM.[10] Metal-coated fiber probes with a nominal aperture of 200 nm (Nanonics Supertips, Israel) made from standard 3M comp. fiber having a diameter of 125 μm were used for the experiments. The SNOM fiber tip was positioned over the flat portion of the cantilever, as shown in Fig. 1. Two optical microscopes and an $x-y$ table made it possible to properly align the fiber tip against the cantilever. When optical shear-force detection[2] was utilized [inset (a) of Fig. 1] the fiber was dithered by a calibrated piezotube, and the fiber tip was illu-

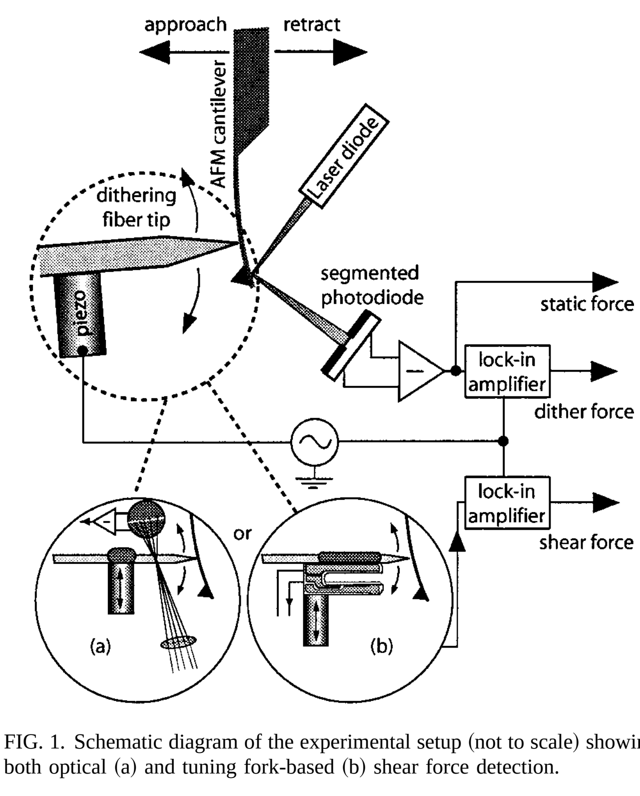

FIG. 1. Schematic diagram of the experimental setup (not to scale) showing both optical (a) and tuning fork-based (b) shear force detection.

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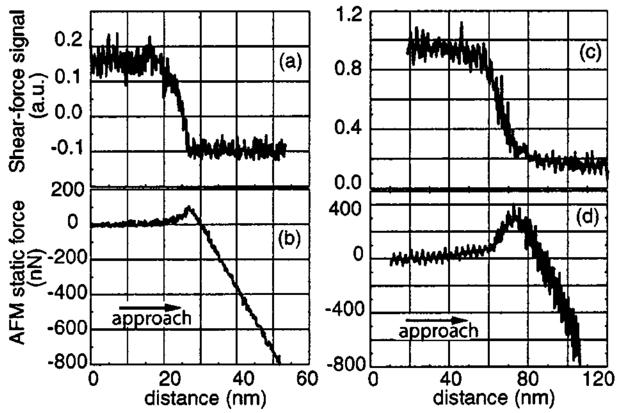

FIG. 2. Shear force (a), (c) and AFM (b), (d) approach curves. (a), (b) Resonantly driven tip ($f=13.8$ kHz, $Q\sim800$), optical detection, dither amplitude - 6 nm; (c), (d) tuning fork detection ($Q\sim500$), dither amplitude - 3.5 nm. The shear force transition looks noisy due to the small time constant of the lock-in amplifier. It was deliberately set low to get a short settling time for the shear force signal to avoid an erroneous time correspondence with the static force signal.

minated with a focused laser diode light beam. Scattered light was detected by a segmented photodiode, and the ac signal at the excitation frequency was converted to a dc signal using a lock-in amplifier (SR850, Stanford Research Instruments). For the tuning fork-based detection scheme [inset (b) of Fig. 1], the fiber was glued with rosin in the usual fashion[4] to one prong of a 32 kHz resonant quartz tuning fork. The fork with the fiber was dithered by the same piezotube, and the piezoelectric signal of the tuning fork at the resonance frequency was converted to a dc signal by the lock-in amplifier. As usual, these dc signals will be referred to as the shear force signals.

The AFM signal was measured by monitoring the deflection of an external laser-diode light beam from the backside of the cantilever with a segmented photodiode.[7] Its output voltage U can be easily converted to an absolute force value $F=k\Delta z$ using the known spring constant of the AFM cantilever k and the displacement Δz of the fiber determined from the voltage applied onto the piezotube. The linear repulsive part of the recorded force curves [see Figs. 2(b) and 2(d)] was used for this force calibration. The spring constant of the fiber is much larger than that of the AFM cantilevers used and thus can be neglected. Two types of noncontact rectangular cantilevers were utilized in our experiments: aluminum-coated silicon cantilevers (NSC12/50, NT-MDT, Moscow, Russia) with nominal spring constant $k_{\mathrm{nom}}=14$ N/m, and uncoated silicon cantilevers (RTESP7, Digital Instruments, Santa Barbara, USA) with $k_{\mathrm{nom}}=40$ N/m. These nominal spring constants were corrected to account for the fact that the force was applied on the cantilever somewhere away from its edge as depicted in Fig. 1. For a rectangular cantilever the force constant is inversely proportional to the cube of its length,[7] and thus the effective spring constant is $k_{\mathrm{eff}}=k_{\mathrm{nom}}(l/l_{\mathrm{contact\text{-}base}})^3$. The value of the ratio $l/l_{\mathrm{contact\text{-}base}}$ was measured via an optical microscope.

Two components of the force can be distinguished in the cantilever deflection signal. The first is a static deflection caused by a static tip–sample interaction; this component is seen in Figs. 2(b) and 2(d). The second is a force component

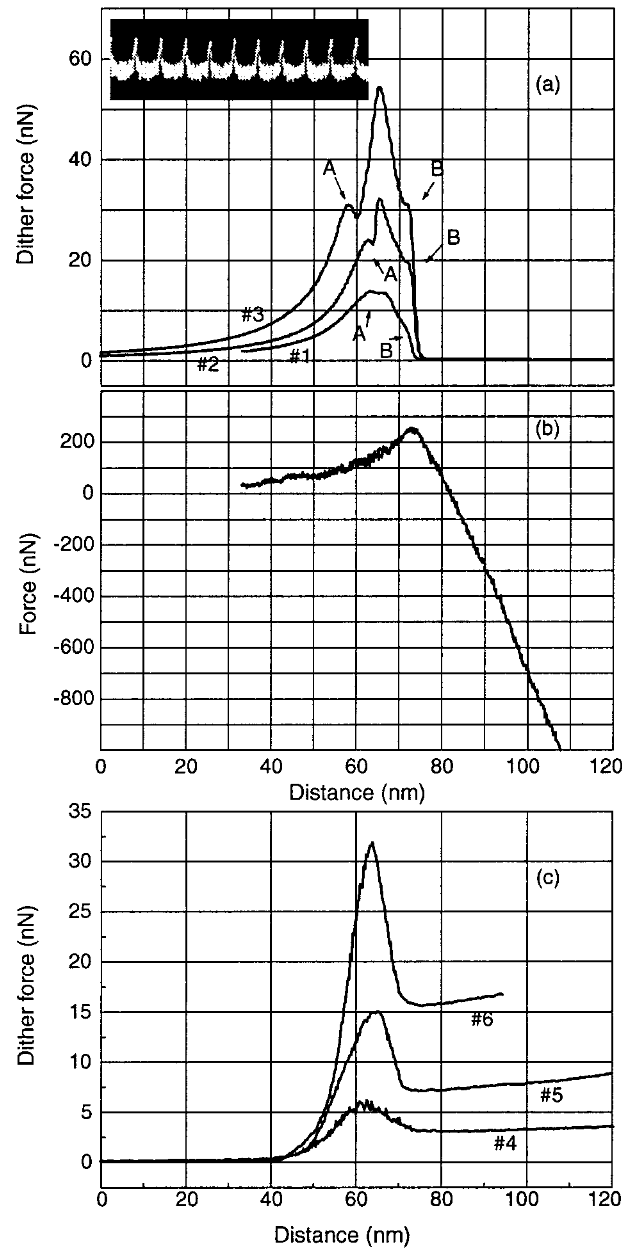

FIG. 3. Approach curves for the dithering force (a), (c) and corresponding cantilever deflection (calibrated in force units); (b) the cantilever deflection curve corresponding to curve (c) here is the one shown in Fig. 2(d). Curves (a) and (b) are recorded for the resonantly driven tip (frequency $f=13.9$ kHz, $Q\sim800$), optical detection; curve (c) for the tuning fork-based detection ($Q\sim500$). Dithering amplitude: No. 1: - 6, No. 2 - 16, No. 3 - 30, No. 4 - 3.5, No. 5 - 14 and No. 6 - 18 nm. In the inset the oscilloscope trace of the AFM cantilever deflection (optical in-resonance detection) shows peaks due to tapping at the dithering frequency of fiber tip.

that oscillates at the dithering frequency of the tip and it was recorded using an additional lock-in amplifier (see Fig. 1). We call this component ''dither force.'' This dither force is well illustrated by the oscilloscope trace in the inset of Fig. 3 recorded for a small drop in the shear force signal (optical in-resonance detection). The shape of this trace is very different from the excitation sine wave which indicates that the tip is only intermittently touching (tapping) the surface, as was discussed in Refs. 3,9,11,12 and others. However, the dither force signal may be observed relatively far from the surface due to electrostatic long-range forces, even when no tapping contact exists.

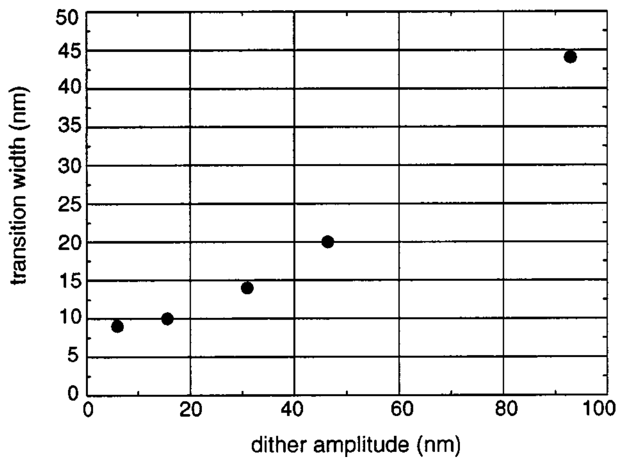

FIG. 4. Dependence of the shear force transition width on the dither amplitude.

From the approach curves presented in Fig. 2 one can see that the shear force signal decreases until contact between the fiber and the lever is established and it is no longer detected in the repulsive part of the AFM force curve. In Fig. 3 we present a set of approach curves for the dither force and the corresponding static AFM signal. Point A in Fig. 3(a) corresponds to the beginning of the shear force transition while point B corresponds to its end. A single pronounced force maximum occurs between A and B. The measurement of distance A–B enabled us to reveal the nonlinear dependence of the width of this transition on the dithering amplitude of the fiber which is presented in Fig. 4. It is easy to see that the shear force transition width is around 9 nm for dither amplitude of 3.5 nm (Ref. 13) when the fiber is tilted 3° with respect to the normal. This value strongly contradicts the 0.2 nm one would expect from the tapping contact model for this geometry,[3] but can be easily explained by the existence of the earlier reported[1,2,4–7,14–16] thin contaminant (water) layer on the sample surface. Upon its approach, the tip first makes contact with a liquid layer, wetting (formation of a "water neck" around the tip) occurs and force is exerted onto the cantilever through this layer. Only upon approaching further does "real" tapping between the tip and the solid surface take place. Thus, for small dither amplitudes the width of the shear force transition is completely determined by the thickness of the adsorbed water layer (cf. Ref. 16) which can be estimated from our experimental data (see Fig. 4) as 9 nm.

The long-range static attractive interaction, which is usually neglected, proved to be very important in our study. The maximal attraction force attains 300 nN [see Fig. 2(d)] and thus essentially exceeds the dither force. Dither force approach curves clearly show that a small attractive force can be detected as far as 100 nm from the surface. At such large distances, as studied in AFM,[15] Coulomb force due to both localized surface charges and a difference in surface potential is by far the largest one. Thus, as is known from AFM practice, in some cases (conductive samples), the total force can be essentially decreased by careful regulation of the tip–sample potential difference. The results of such a procedure are illustrated by approach curves in Fig. 3(c), which were recorded using a cantilever coated with a 10 nm thick layer of platinum. The curves no longer reveal long electrostatic tails, thus supporting our assumption. However, a "bump" of the same order of the magnitude still exists on the AFM force curve inside the range of shear force transition. It is not usually observed in atomic force microscopy with standard cantilevers as stiff as the ones used in the present work. This bump is caused by the large radius of curvature (100–500 nm) of the SNOM fiber tips compared to the typical value of 5–10 nm for AFM cantilever tips.

TABLE I. Measured values of the interaction force.

Detection method		Optical in resonance			Tuning fork	
Dither amplitude (nm)	6	16	30	3.5	14	18
Dither force (nN)	13	32	55	6	15	32
Tilt angle (g rad)		$3° \pm 1°$			$2° \pm 1°$	

From the data recorded we determine the dither force acting during the shear force-based fiber probe SNOM experiments. Obviously, this value depends on the choice of setpoint installed for feedback regulation. To avoid any ambiguity, in Table I we present the maximal value of the shear force occurring during the shear force transition. A dither force of the order of 1 nN can be easily achieved. Further reduction is possible by applying subnanometer dither amplitudes and careful choice of the operating setpoint and the increase of the Q factor. This may make it possible to reduce the force to a value of the order of ~ 10 pN.[14] However, in general, this makes sense only if the static interaction is also reduced.

This work was supported by the Swiss National Science Foundation, Grant No. 2000-065160.01, and by SCOPES.

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