

# Piezoelectric tip-sample distance control for near field optical microscopes

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An aluminum coated tapered optical fiber is rigidly attached to one of the prongs of a high  $Q$  piezoelectric tuning fork. The fork is mechanically dithered at its resonance frequency (33 kHz) so that the tip amplitude does not exceed 0.4 nm. A corresponding piezoelectric signal is measured on electrodes appropriately placed on the prongs. As the tip approaches within 20 nm above the sample surface a 0.1 nN drag force acting on the tip causes the signal to reduce. This signal is used to position the optical fiber tip to about 0 to 25 nm above the sample. Shear forces resulting from the tip-sample interaction can be quantitatively deduced. © 1995 American Institute of Physics.

In a near field scanning optical microscope (NSOM), an optical aperture with subwavelength sized diameter  $\phi$  is scanned in the direct vicinity of a sample surface.  $\phi$  is typically of the order of 50 to 200 nm. The aperture generally illuminates locally the sample surface and optical information with spatial resolution of the order of  $\phi$  is accessible.<sup>1,2</sup> Optimal spatial resolution is reached when the tip is at distances smaller than  $\phi$  away from the surface.<sup>2</sup> Because NSOMs apertures are scanned in such near field proximity of surfaces it is necessary to prevent tip-sample catastrophic collisions. For this reason, tip-sample distance regulation plays a central role in NSOMs. To date, the distance control most commonly used in NSOMs are based on optical detection of shear forces acting on the tip.<sup>3-5</sup> In such detection methods, the tip is usually vibrated in a motion parallel to the sample surface at one of its mechanical resonances. As it is approached perpendicularly to and about few tens of nanometers above the sample surface, the amplitude of the tip vibration (typically, 5 to 10 nm) decreases. The nature of such shear forces is to the best of our knowledge still unclear. Optical detection of the tip vibration amplitude can be performed by differential interferometry<sup>3</sup> or by measuring the oscillation in the specularly reflected intensity of a laser beam focused on the tip end.<sup>4,5</sup> Although such methods are proven to allow reliable operation in friendly experimental conditions, they do not appear to be convenient for operation at low temperatures, in high vacuum conditions, or in situations where samples space sizes are restricted.<sup>5</sup> Furthermore, in cases where visible and infrared background photon intensity should be limited to that of the tip aperture such optical detection cannot be used. We present in this letter an alternative nonoptical method based on a piezoelectric detection of tip-sample interaction.

In the present design, we take advantage of the mechanical resonance of a piezoelectric tuning fork with large quality of factor  $Q$ . As shown in Fig. 1, the optical fiber with its aluminized tapered tip is glued along the side of one of the prongs of a quartz crystal tuning fork. Such tuning forks are commercially available for operation at 32 768 Hz (i.e.,

2<sup>15</sup>). In order to excite the mechanical resonance of the fork, it is rigidly mounted on a ceramic piezoelectric tube serving the purpose of a dither. The tuning fork and the tip are vibrated parallel to the sample surface. Both prongs are piezoelectrically coupled through the contact pads A and B shown in Fig. 1. On resonance, the bending amplitude of the prongs is maximum. This in turn generates an oscillating piezoelectric potential proportional to the tip oscillation amplitude. A typical resonance of the piezoelectric signal amplitude is shown in Fig. 2. When used for feedback, the fork is driven at resonance. This oscillating signal, picked up between both contacts and measured using a lock-in detection synchronous with the dither frequency, is monitored as the tip approaches normally onto the sample plane. Similarly to what happens with optical detection, a reduction of the signal amplitude is measured as the tip is within tens of nm of a sample surface. This change in signal is shown in Fig. 3. Such piezoelectric signals can be used in conjunction with an electronic feedback loop for surface topography imaging as demonstrated in Fig. 4. In the rest of this letter we will show that this device

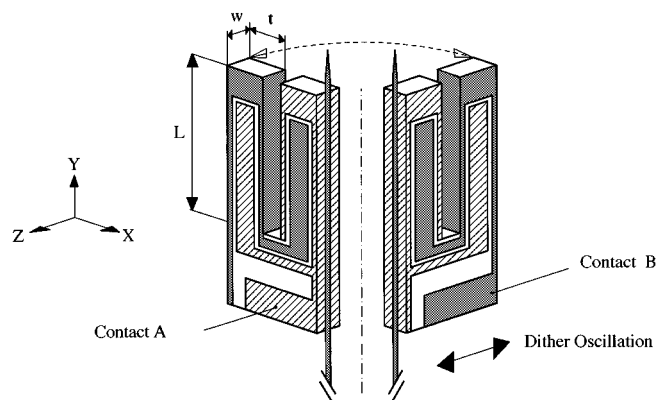


FIG. 1. Front and back view of a crystal quartz tuning fork with a 125  $\mu$ m tapered aluminum coated fiber probe glued along one of its prongs. X, Y, and Z are the quartz crystal axis. The dither vibrates the whole device along X. The shaded and dark areas represent both contact pads serving both as pickup for the piezoelectric signal as well as coupling between the two prongs.  $L=4$  mm,  $t=0.6$  mm,  $w=0.4$  mm. The tip protrudes 0.8 mm out of the prong's end.

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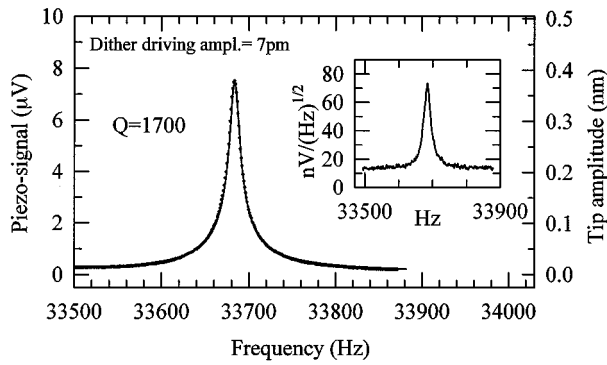


FIG. 2. Amplitude of the piezoelectric signal as function of the driving frequency. The diether driving the fork vibrates with an amplitude of 7 pm. Points are measured. The full line is calculated using the harmonic oscillator model described in text with  $Q$  as a fit parameter. The tip amplitude on the right scale is obtained by differential interferometric measurements. Inset: noise signal, for the freely oscillating fork.

allows to measure quantitatively the amplitude of the shear force originating from the tip-sample interaction.

The prong holding the tip is a cantilever of length  $L$  and rectangular cross-section  $wt$ . It bends periodically along the  $X$  direction of Fig. 1. Mechanical properties of elastically vibrating homogenous cantilever are analytically derived in textbooks such as Ref. 6. The parameters needed to determine the dynamics of the prong are its dimensions  $L$ ,  $w$ ,  $t$ , its Young modulus  $E$ , and its density  $\rho$ . Within this model,<sup>6</sup> the time dependence of the tip position  $x(t)$ , turns out to be described by an effective harmonic oscillator equation of motion driven at frequency  $\omega$ :  $m_e \partial^2 x / \partial t^2 + F_D + kx = Fe^{i\omega t}$ , where  $m_e = 0.2427\rho(Ltw)$  is an effective mass corresponding to about 1/4 of the mass of one of the prongs.  $F$  is the amplitude of the driving force tuned by adjusting the voltage applied on the dither piezotube.  $k = Ewt^3 / (4L^3)$  is the static compliance of one prong; and  $F_D = m_e \gamma \partial x / \partial t$  is a phenomenological viscous force which is the sum of all drag forces acting on the cantilever. In the present model, we will assume that the shear force resulting from the tip-sample interaction, is in fact a drag force contributing to  $F_D$ . This assumption is reasonable since the tip is in frictional motion

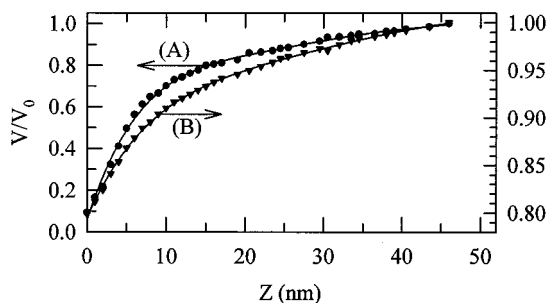


FIG. 3. Approach curves of the normalized signal amplitude (at resonance frequency) as function of the tip-sample distance. The signal in curve A, results from an optical measurement of the amplitude of a resonance mode of the tip as described in Ref. 5. Curve B is the simultaneous measured piezoelectric signal of the tuning fork with a  $Q=200$ . Lines are a guide to the eye.

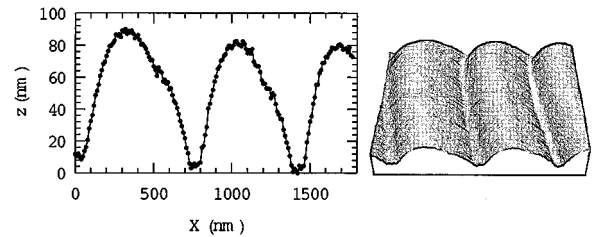


FIG. 4. Topography measurement using the on resonance piezoelectric signal as a feedback signal controlling the tip-sample distance at about 5 nm above the surface. The sample is a grating of developed photoresist exposed in an laser interference pattern.  $1.8 \times 1.2 \mu\text{m}$  image acquired in 200 s.

parallel to the sample plane. For small drag (i.e.,  $\gamma \ll \omega$ ), the solution for the above differential equation is  $x = (F/m_e) / (\omega_0^2 - \omega^2 + i\gamma\omega) e^{i\omega t}$  giving in turn  $F_D = im_e \gamma \omega x$ . Both  $x$  and  $F_D$  show a resonance at frequency  $\omega_0 = (k/m_e)^{1/2} = 2\pi f_0 = 1.0150t/L^2(E/\rho)^{1/2}$ . As shown in Fig. 2, the Lorentzian dependence of the amplitude of  $x$  on  $\omega$  predicted by this model is in excellent agreement with the measurements. On resonance, the tip amplitude is  $x_0 = FQ\sqrt{3}/ik$  and the drag force experienced by the fork is given by<sup>8</sup>  $F_D = ikx_0/(Q\sqrt{3})$ . The quality factor is defined as  $Q = f_0/\Delta f$ , where  $\Delta f$  is the full frequency width at half-maximum of the tuning fork amplitude resonance. In order to evaluate  $|F_D|$  we need to determine  $k_0$ ,  $x_0$  and  $Q$ . Since the exact amplitude of the driving force  $F$  is generally unknown,  $x_0$  has to be determined in an independent way. We have measured  $x_0$  optically using a Normarski differential interferometer. Using this technique, the on resonance piezoelectric signal  $V_0$  is calibrated against  $x_0$ . This calibration is done once for all forks of same geometry. It is, typically, in our case  $V_0/x_0 = 27 \mu\text{V}/\text{nm}$ . We normally operate our tip-sample distance regulation using  $x_0 = 0.35 \text{ nm}$ . In principle,  $V_0$  can be determined from the fork parameters it is only relevant to mention that  $V_0 \approx d_{12} L k x_0 / t^2$ , where  $d_{12} = 2.31 \times 10^{-12} \text{ V/m}$  (or  $\text{C/N}$ ) is the piezoelectric coupling coefficient for quartz. The static compliance  $k = 26600 \text{ N/m}$  is determined using  $E = 7.87 \times 10^{10} \text{ N/m}^2$  along the  $x$  or  $y$  quartz crystal direction,  $L = 4 \text{ mm}$ ,  $t = 0.6 \text{ mm}$ ,  $w = 0.4 \text{ mm}$ .<sup>7</sup> Alternatively,  $k$  can also be independently inferred from  $k = m_e \omega_0^2$ . Using  $f_0 = 32768 \text{ Hz}$  and  $\rho = 2650 \text{ kg/m}^3$ , we find  $k = 26200 \text{ N/m}$  which is in good agreement with the former value.  $Q$  is obtained by measuring the full frequency width  $\Delta f$  measured at half-maximum of the piezoelectric signal amplitude resonance. A bare tuning fork (i.e., with no optical fiber glued on it), has at room temperature a  $Q = 64200$  in vacuum and 7500 in air. When an optical fiber is glued along one of the prongs,  $Q$  is usually reduced to about 1000.  $Q$ 's as high as 3650 were nevertheless obtained. In absence of tip sample interaction contact, the drag force originates mostly from losses internal to the fork/fiber system. Using  $Q = 1000$  and  $|x_0| = 0.35 \text{ nm}$ , we evaluate  $|F_D| = 5.4 \text{ nN}$ . When the tip is in the interaction range of the sample,  $x_0$  and the  $Q$  reduce in same proportions. This is because  $|x_0| = \sqrt{3}FQ/k$ . In other words on interaction,  $x_0$  and  $Q$  conspire to reduce proportionally keeping  $F_D$  constant. However, in this situation, part of  $F_D$  is now due to the

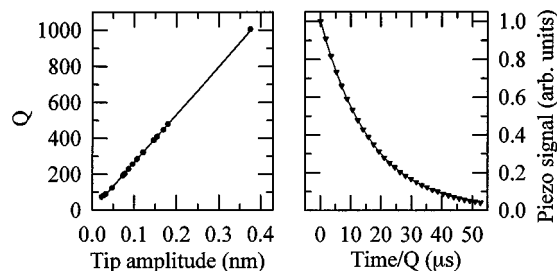


FIG. 5. Left panel:  $Q$  and tip amplitude are measured independently showing the linearity dependence predicted by the effective harmonic oscillator model (line). Right panel: piezoelectric signal amplitude relaxation of a freely oscillating tuning fork measured as function of time normalized to  $Q$  (triangles). The solid line is calculated (not fitted) using the independently measured value for  $Q$ . For  $Q=1000$  the signal relaxes exponentially with  $\tau=18$  ms. This limits the maximum scanning speed of the tip across a sample surface.

tip/sample interaction. It is then easy to show that the tip-sample contribution to the drag is simply

$$|F_S| = |F_D|(1 - V/V_0), \quad \text{where} \quad |F_D| = k|x_0|/(Q\sqrt{3}), \quad (1)$$

where  $V$  is the piezoelectric signal amplitude during tip-sample interaction, and  $V/V_0$  is plotted in Fig. 3. In Eq. (1)  $x_0$  and  $Q$  are the values measured with the tip away from sample. Using the above parameters values we determine that a change of 1% in the signal corresponds to  $|F_S| = 54$  pN. We have recorded that  $F_S$  acting on the tip can be up to 2 nN without tip aperture damage and up to 40 nN before tip damage. In normal operation, we take NSOM and topography images with  $F_S$  ranging between 50 and 500 pN.

We have tested several aspects of the relevance of the effective harmonic oscillator model. To do so we have performed hydrodynamic measurements of viscous drag in a controlled case. A 125  $\mu\text{m}$  flat cleaved cylindrical optical fiber is attached to the side of one of the prongs. 1 mm of the fiber is protruding outside the prong. The protruding part of the fiber is dipped with a depth  $d$  into a fluid of known viscosity. We have chosen this geometry because the viscous drag of a cylinder moving in a liquid is known and has an analytical form for small oscillation amplitude.<sup>9</sup> At constant drive  $F$ , the larger is  $d$  the larger is the viscous drag resulting in a smaller  $Q$  and  $x_0$ . By adjusting  $d$ , we can tune the drag. This permitted for instance to check the proportional dependence of  $Q$  with  $x_0$  as shown in Fig. 5. It is also found that as  $Q$  drops from 1000 to 500,  $f_0$  does not shift more than 0.5 Hz. Using a simple hydrodynamic model,<sup>9</sup> the viscous drag detected by the tuning fork is now independently evaluated<sup>10</sup> and is found to be within 20% in agreement with the result given by Eq. (1). We also verified that as  $d$  increases,  $x_0$  and  $Q$  change in a way that keeps the hydrodynamical measured  $F_D$  constant. The fact that the tuning fork can be oscillated with high  $Q$ 's is what is making such drag detectors sensitive to velocity dependent forces of the order of 50 pN. This is because on resonance the cantilever has an effective compliance of  $k_{\text{eff}} = k/(Q\sqrt{3})$ . On the other hand, high  $Q$ 's prevent from scanning rapidly. High  $Q$ 's prevent, from scanning rapidly. The simple harmonic oscillator

model predicts that when the fork is displaced from equilibrium, the prong's amplitude relaxes exponentially with a rate  $\tau = \sqrt{3}/(\pi\Delta f)$ . As shown in Fig. 5, we have measured the time dependence of  $x_0$  as the fork relaxes by setting  $F=0$  at  $t=0$ . Using  $\Delta f=30$  Hz, measured independently, we determine  $\tau=18$  ms. The calculated time dependence of the amplitude relaxation is in agreement with the observation to better than 1%. This analysis shows that it is desirable to reduce  $Q$  but also  $k$  or  $x_0$  in order to allow for more rapid scanning. There is a room for improvement in terms of reducing  $k$ . Preliminary measurements using tuning forks with softer prongs (i.e.,  $k=2400$  N/m<sup>2</sup>) and  $Q=100$  show promising results. In the present arrangement, the noise on the piezoelectric signal corresponds to a rms amplitude of about  $\langle \delta x^2 \rangle^{1/2} = 20$  pm in a bandwidth of  $1/\tau$ . It is essentially limited at the present time by mechanical and electrical pickup noise. This noise figure could be in principle improved to the noise limit defined by the thermal excitation  $K_B T$  of the prongs. For a one-dimensional harmonic oscillator the equipartition theorem dictates  $\langle \delta x^2 \rangle = K_B T/k$  (on resonance) over a self-limited bandwidth of  $1/\tau$ . At temperature  $T=300$  K, the floor noise should be  $\langle \delta x^2 \rangle^{1/2} = 0.4$  pm.

In conclusion, we have presented here a body of evidence showing that the piezoelectric tuning fork presents an attractive alternative for distance control feedback methods. This is specially the case in the light of the fact that preliminary measurements show that this feedback system operates at 2 K and under high fields and that improvement of a factor 10 both in sensitivity and speed can be achieved.

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- <sup>5</sup>R. D. Grober, T. D. Harris, J. K. Trautman, and E. Betzig, *Rev. Sci. Instrum.* **65**, 626 (1994) and references therein.
- <sup>6</sup>D. Sarid, in *Scanning Force Microscopy* (Oxford University Press, New York, 1991), Chap. 1.
- <sup>7</sup>At this point is worth mentioning that the length of the optical fiber glued on the prong should increase  $k$ . For the present case this effect contributes, however, to a compliance enhancement of 0.2%.
- <sup>8</sup>The appearance of  $\sqrt{3}$  in the various terms above comes from the definition of the quality factor is defined not for an energy as usually done but rather for an amplitude.
- <sup>9</sup>See, e.g., R. Beker, in *Fluid Dynamics II*, Handbuch der Physik, Vol. VIII/2 (Springer, Berlin, 1963), p. 1.
- <sup>10</sup>The only effect we did not appropriately account for is that of the liquid surface tension acting on the walls of the cylinder. In oil, this effect turns out to be small for  $d$  larger than 40  $\mu\text{m}$ .