Tip-sample distance control for near-field scanning optical microscopes

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ABSTRACT

In a recent paper (Karrai and Grober Appl. Phys. Lett. 66, 1842, (1995)) we have developed a new technique allowing to control the distance separation between a tapered metal coated optical fiber tip and the surface of a sample. This technique is based on a piezo-electric tuning fork used as a shear force sensor and is found to be suitable for the operation of near-field scanning optical microscopes. We present in this article a review of the recent results concerning this new technique. In particular, we present hydrodynamic measurements performed in order to evaluate the shear (or viscous drag) forces picked up by the piezo-electric tuning fork sensor.

1. INTRODUCTION: THE PIEZO-ELECTRIC TUNING FORK SHEAR FORCE DETECTOR:

One of the important component of a near-field scanning optical microscope (NSOM), is the feedback distance control that maintains the tip at constant height above the sample\textsuperscript{14}. NSOM design specifications require that such a tip sample distance control should be achieved with precision of the order of the fraction of nanometer. In a recent paper\textsuperscript{1} we have proposed and demonstrated the operation of a new tip-sample distance control device based on a resonant piezo-electric tuning fork used as a shear force detector. In this introduction we review the operation of such a feedback mechanism described in ref.1.

The idea behind such a shear force detector, is to take advantage of the mechanical resonance of a piezoelectric tuning fork with large quality factor Q. Fig. 1 shows the optical fiber with its aluminized tapered tip glued along the side of one of the arms of a quartz crystal tuning fork. The tip at the end of the optical fiber protrudes out of the arm on which it is attached. The tuning fork is used as a force sensor, while the tip acts as a shear force pick-up. The fork is rigidly attached onto an external piezo-electric vibrator (i.e. the dither). The purpose of the dither is to excite the tuning fork on its mechanical resonance. The tuning fork is vibrated in such a way that the tip oscillates parallel to the sample surface. Both arms of the fork are piezo-electrically coupled through the metallic contact pads A and B shown in fig.1. The geometry of the contacts and the coupling between the two arms insures that only one resonant vibration mode of the fork is excited. On resonance, the bending amplitude of the prongs is maximum. This in turn generates an oscillating piezo-electric potential proportional to the tip oscillation amplitude.

Fig.1: Front and back view of a crystal quartz tuning fork with a tapered aluminum coated optical fiber probe attached along one of its arms. X, Y and Z shows the crystal quartz axis. The dither vibrates the whole device along X. The shaded areas shows both metal contact pads which used to pick up the piezo-electric signal as well as to couple the motion both arms in a tuning fork type of oscillator.

A typical resonance of the piezo-electric signal amplitude is shown in fig.2. When used for feedback tip-sample distance control, the fork is driven at its resonance frequency. The resulting oscillating piezo-electric signal, picked up between both contacts is monitored as the tip approaches normally onto the sample plane. A reduction of the piezo electric signal amplitude is measured as the tip is within tens of nanometers.
Fig. 2: Amplitude of the piezo-electric signal of a tuning fork (mounted with an optical fiber tip) as a function of the dither driving frequency. The dither vibrates the whole tuning fork and the optical fiber tip with an amplitude of 7 pm. The points are the measured data and the full line is calculated using the effective harmonic oscillator model described in the text.

Fig. 3: Approach curve showing the piezo-electric signal reduction as a function of the tip sample distance. The arrows indicates the direction of the tip sample approach. The shear force amplitude indicated on the left scale was obtained using equation (35) with the measured $Q_{\text{v}}=1000$, $v_{\text{sf}}=0.4$nm and the calculated $k_{\text{int}}=26 \mu$N/nm.

of a sample surface. Such a signal reduction is measured when the tip is within distances of the order of 40 nm from the sample as shown in fig. 3. The piezo electric signal is used in conjunction with an electronic feedback loop maintaining the tip at constant shear force interaction allowing for surface topography imaging as demonstrated in ref. 1. The schematic operation of the NSOM microscope in conjunction with the quartz-tuning fork tip-sample distance control is shown in fig. 4. In the following sections 2 through 6 we review the elementary idealized model analysis leading to the design parameters for piezo-electric tuning fork shear force detectors. Such an analysis is based on classical text book physics on cantilever elastic deformation and piezoelectricity.

In paragraph 7 the results of a hydrodynamic experiment are presented giving an evaluation of the strength of shear forces picked-up by this detector. In paragraph 8, examples of friction force images taken with tapered aluminum coated optical fiber tips are presented. In the last paragraph we show that, the fork can be self vibrated, without the need for an external dither, while retaining its force sensing capabilities.

Fig. 4: Schematic view of the NSOM reflection microscope using a piezo-electric quartz tuning fork for the tip-sample distance control. In this mode, the oscillator drives the piezo-electric dither with a typical amplitude of 0.01nm. The frequency of the oscillator is tuned with that of the resonance frequency of the tuning fork (typically 33kHz). When the tuning fork is vibrated on its resonance, its arms near the optical fiber tip oscillate with an amplitude of typically of 5nm. The oscillation of the tuning fork arms induces a piezoelectric signal proportional to the arms oscillation amplitude. This signal is detected with a lock-in detector synchro with oscillator voltage driving the dither. The amplitude of the detected signal measured with a typical time constant of 1msec is feed to a feedback electronic loop comparing this signal with a set signal. When the tip approaches the sample surface to within an interaction region (typically 10nm), tip-sample friction forces damps the tip-tuning fork oscillation amplitude. This in turn leads to a reduction of the amplitude of the measured piezo-electric signal picked-up at the forks metallic contact pads. The digital signal processor (DSP) control keeps the tip within a constant shear force interaction with the sample surface. This way, a friction force microscopy (FFM) image is obtained simultaneously with a reflection NSOM image.
2. THE VIBRATING CANTILEVER MODEL:

The arm of the tuning fork holding the tip is a cantilever of length $L$, thickness $T$ and width $W$ as indicated in fig.1. It bends periodically along the $X$ direction of fig.1, and is assumed to be rigidly anchored in the plane $y=0$. We will assume that the protruding tip is short enough so that it can be considered infinitely rigid, acting therefore as a perfect shear force pick-up along $X$. We assume also that the tip position lies in the plane $y=L$. Finally we will assume that the optical fiber part glued on the arm is soft enough that it does not contribute significantly to the compliance of the cantilever.

If a force $F$ is applied along $X$ at $y=L$, the arm will bend correspondingly along $X$. The bending radius $R(y)$ of the long axis (i.e. the neutral stress axis) of the cantilever is given at position $y$ on the $Y$ axis by

$$1/R(y)=M(y)/(EI)$$

where $M(y)$ is the bending moment, $E$ is the elasticity modulus (i.e. Young's modulus) of the piezo electric fork material, and $I=x^2dx$ is the moment of inertia of one arm. In the case of homogeneous cantilevers with a rectangular cross section $WT$, the moment of inertia is $I=WT^3/12$. For small bending amplitude, we can write $1/R=\partial^2u/\partial y^2$ where $u(y)$ is the deformation of the cantilever defined as the displacement of the arm's long axis away from the its rest axis $Y$. In this limit eq. 1 reduces to

$$\partial^2u/\partial y^2=\frac{M(y)}{(EI)} \tag{2}$$

In the case of static deformation, the bending momentum is solely due to $F$ and is $M(y)=F(L-y)$. In this case the static deformation $u(y)$ is trivially obtained by solving eq. 2 giving:

$$u(y)=[2y^3(3L-y)/(EWT^3)] F \tag{3a}$$

$$u(L)=u_L=4L^3/(EWT^3) F \tag{3b}$$

$$u(y)=[y^3(3L-y)/(2L^3)] u_L \tag{3c}$$

were eq. 3c is a formulation of the deformation in which the applied force does not enter explicitly. The displacement $u_L$ of the end of the cantilever corresponding also to the tip position is given for $y=L$ by equation 3b as a function of the applied bending force $F$. Eq. 3b shows that the arm acts as a spring with a spring constant $k_{sun}$ reacting with a force $F=k_{sun}u_L$ to a displacement $u_L$ away from equilibrium. This gives the first relevant design formula namely the static spring constant of the tuning fork arms:

$$k_{sun}=(E/4) W (T/L)^3 \tag{4}$$

In order to obtain the dynamical properties of the cantilever, such as its resonant frequency excitation (i.e. the tuning fork resonance frequency), we need first to establish the equation of motion of an infinitessimal thin slice $dy$ of the arm. The Newton equation of motion for this element writes:

$$\delta t=(\tau+\delta t)\tau=\delta m(\partial^2u/\partial t^2) \tag{5}$$

where $-\tau$ and $+\delta t$ are the shear forces oriented along $X$ experienced by the cantilever in the planes $y$ and $y+dy$ respectively. $\delta m=\rho WT dy$ is the mass of the arm element and $\rho$ is the fork material density. This equation is supplemented by the condition that at any time the sum of all moments applied on the cantilever elements $dy$ should add to zero. This condition is:

$$(M+\delta M)-(\tau+\delta t)dy+\delta t\delta y/2=0 \tag{6}$$

As seen from eq. 5, $\delta t$ is proportional to $\delta y$, consequently, the last term of eq. 6 can be ignored to the first order of $\delta y$. Eq. 6 reduces then to $\tau=\partial M/\partial y$ in the limit of $\delta y=0$. This expression of $\tau$ together with the $\delta y=0$ limit of equation 5 gives the new equation of motion

$$\partial^2 M/\partial y^2+\rho WT (\partial^2u/\partial t^2)=0 \tag{7}$$

Making use of eq. 2 which relates $u$ to $M$ we are left with the classical equation of motion for vibrating levers

$$\partial^2 u/\partial y^4+\rho WT/(EI) (\partial^2u/\partial t^2)=0 \tag{8}$$

We specialize from now on the analysis to solutions of eq.8 having a periodic time dependence $u=u_0e^{i(\omega t)}$, where $\omega_0=2\pi f_0$ is one of the normal mode resonance frequency of the arm. In this case eq. 8 reduces to the resonant lever equation of motion

$$\partial^4 u/\partial y^4=(\omega_0^2) u \tag{9a}$$

$$\omega_0^2=\rho WT/(EI) \omega_n^2 \tag{9b}$$

he differential eq.9a is to be solved with the boundary conditions $u(0)=0$ and $\partial u/\partial y=0$ indicating that the arm is anchored at its basis. For a free vibrating resonator the supplemental boundary conditions $M(L)=0$ and $u(L)=0$ are implied. This in conjunction with equation 2 implies that $[\partial^2u/\partial y^2]_{y=L}=0$. Furthermore, since $\tau=\partial M/\partial y$ the last boundary condition is $[\partial^2u/\partial y^2]_{y=L}=0$. The general solution of eq.9 with this four boundary conditions leads to the eigenvalue equation for $\omega_n$.

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\[ \cos(\alpha L) \cosh(\alpha L) + 1 = 0 \]  

The fundamental and the first 5 harmonic values of \( \alpha L \) satisfying eq.10 are:
\[ \alpha L = \eta_i = 1.875, 10, \alpha L = \eta_i = 4.69409, \alpha L = \eta_i = 7.85476, \alpha L = \eta_i = 10.99554, \alpha L = \eta_i = 14.13717, \alpha L = \eta_i = 17.27876. \]

For larger values of \( n \), the eigen mode solutions satisfy \( \alpha L = \eta_i = (n+1/2) \pi \). Making use of the definition of \( \alpha_n \), given in eq.9b, and specializing to the case of rectangular levers for which \( I = wT^2/12 \), the resonance frequencies are:

\[ \alpha_n = 2\pi f_n = (T/L^2)(E/12\rho)^{1/2} \]  

\[ \alpha_0 = 2\pi f_0 = 1.0150/(T/L^2)(E/\rho)^{1/2} \]  

Eq.11b is the second relevant design formula for the fundamental resonant frequency mode for the tuning fork. In a realistic situation in which an optical fiber is glued along the arm, the above formula are only approximate. The fiber will contribute to make the cantilever stiffer and the resonant frequencies are expected to be somewhat larger than the one given by equation 11b.

As we will see it in the following section, the sensitivity of the fork is determined mostly by its spring constant \( k_{\text{st}} \). In order to design the optimum force detector it is desired to specify in first place both the desired specifications \( k_{\text{st}} \) and \( f_0 \) and then deduce the corresponding sizes \( L, T \) and \( W \) of the arms. Since a tuning fork is manufactured by etching it out of a thin quartz wafer of thickness \( W \), it is practical to use \( W \) as a specified parameter as well. Solving the system of eq.4 and 11b for \( T \) and \( L \) we obtain the following tuning fork designer formulae:

\[ L = (1.0150/2\pi f_0) (4k_{\text{st}}/EW)^{1/2} (E/\rho)^{1/2} \]  

\[ T = (1.0150/2\pi f_0) (4k_{\text{st}}/EW)^{1/2} (E/\rho)^{1/2} \]  

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<th>L (mm)</th>
<th>T (mm)</th>
<th>W (mm)</th>
<th>( f_0 ) (Hz)</th>
<th>( k_{\text{st}} ) (( \mu )N/mm)</th>
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Table 1: Sumarize the design values for commercially available quartz tuning fork. For quartz single crystal the following parameters were used: the density is \( \rho = 2659 \text{kg/cm}^3 \) and Young's modulus is \( E = 7.87 \times 10^{10} \text{N/m}^2 \).

3. The Effective Mass Harmonic Oscillator Model:

When driven at its resonance frequency, the arms of the tuning fork oscillates in resonance along \( X \). In this section we calculate the corresponding tip position when the fork is mechanically excited with the dither. The rigorous approach to this problem would be to establish the driven equation of motion version of eq.8 with in addition a lossy force term accounting for energy absorption within the device. Two of such equations should be written for each of the fork arms. Both equation should be related to each other by a piezoelectric coupling force term making the fork operate on just one resonance mode. We did not take this route since, as we will see it, the dynamics of the device can be described phenomenologically in very satisfactory way with a much simpler effective one dimensional harmonic oscillator model. For small amplitudes \( u_x(t) \), each arm of the tuning fork can be thought of as a spring with constant \( k_{\text{st}} \). Consequently, \( u_x(t) \) is the solution of an effective harmonic oscillator equation of motion driven at frequency \( \omega \). The equation of motion for such an oscillator is then simply:

\[ m_0 \ddot{u}_x + F_D + k_{\text{st}} u_x = F \exp(i\omega t) \]  

where \( m_0 \) is an effective mass to be determined and \( F \) is the mechanical driving force imposed by the dither. \( F \) is usually tuned by adjusting the driving voltage on the dither piezo. \( F_D \) is a lossy term proportional to the velocity. It represents a drag force opposing the movement of the fork arms. We will assume that this drag force is the sum of the forces resulting from the tip sample shear force interaction and from an internal drag force corresponding to the viscous losses occurring in the tuning fork arm. \( F_D \) can be entirely characterized by a phenomenological parameter \( \gamma \) that has the dimensions of a frequency if it is written as:

\[ F_D = m_0 \gamma \frac{d u_x}{d t} \]  

The effective mass \( m_0 \) is chosen in a way that the resonant frequency \( f_0 \) of this harmonic oscillator is compatible with the spring constant \( k_{\text{st}} \). This condition is fulfilled by writing:

\[ 2\pi f_0 = \omega_0 = (k_{\text{st}}/m_0)^{1/2}. \]  

Replacing \( k_{\text{st}} \) by its expression in eq.4 and using eq.11b for \( 2\pi f_0 \) the effective is given by:

\[ m_0 = 3m/\eta_0^4 = 0.2427 m \]  

where \( m = \rho (LTW) \) is the mass of one arm of the fork. The equation of motion 13 is trivially solved in the limit of small drag force. This limit corresponds the realistic condition \( \gamma \sim \omega_0 \). In this case, the time dependence of \( u_x(t) \) is simply \( u_x \exp(i\omega t) \). From eq. 13 we get:
\[ u_\gamma(t) = \exp(i\omega t)(F/m_0)/(\omega^2 - \omega_0^2 + i\gamma) \]  

Using eq. 14 the drag force is in turn

\[ F_\gamma(t) = m_0\gamma u_\gamma(t) \]  

Both \( u_\gamma(t) \) and \( F_\gamma(t) \) show a resonance peaked at frequency \( \omega_0 \). The amplitude of \( u_\gamma(t) \) is given by:

\[ |u_\gamma|/|u_\gamma|_{\text{res}} = 2/\sqrt{\eta^2 + (\omega_0^2 - \omega^2)^2} \]  

Where \( u_\gamma = F/m_0/\sqrt{(i\gamma m_0)} \) is the tip oscillation amplitude on resonance (i.e. at \( \omega = \omega_0 \)). The above equation shows that the oscillation amplitude of the tip is a Lorenzian shaped peak function of frequency with a maximum at \( \omega = \omega_0 \). This is a result which was experimentally verified to great accuracy in ref. 1. A Lorenzian shaped resonance can be entirely characterized by its frequency width and its maximum amplitude. At this point we define the following quality factor the so called Q-factor of the tuning fork:

\[ Q_0 = f_0/\Delta f \]  

where \( \Delta f \) is the full width at half maximum (FWHM) of the Lorenzian shaped resonance of the tip oscillation amplitude. This definition is practical since \( \Delta f \) and \( f_0 \) (unlike \( \gamma \)) are directly measurable quantities. 2 From this definition, \( \gamma \) is now related to \( \Delta f \) by solving \( |u_\gamma|/|u_\gamma|_{\text{res}} = 1/2 \) for \( \omega \). The condition \( |u_\gamma|/|u_\gamma|_{\text{res}} = 1/2 \) is the equation giving the frequencies at FWHM. The results is:

\[ \gamma = 2\pi \Delta f / \sqrt{3} \]  

On resonance the tip amplitude and the drag force experienced by the tuning fork is given for \( \omega = \omega_0 \) by

\[ u_{\text{Lo}} = \sqrt{3}Q_0/(i\eta m_0) F \]  

\[ F_{\text{Lo}} = i \eta m_0/(\sqrt{3}Q_0) u_{\text{Lo}} \]  

Note that \( u_{\text{Lo}} \) and \( F_{\text{Lo}} \) are \( \pi/2 \) out of phase. Equation 22a shows that for small losses (i.e. \( \gamma = \omega_0 \)), the maximum vibration amplitude \( u_{\text{Lo}} \) is proportional to \( Q_0 \). This was checked to be experimentally the case for a real piezo electric quartz tuning forks. 1 Equation 22b gives the drag force sensed by the fork as a function of the tip amplitude and does not explicitly depends on the drive force \( F \). Eq. 22b demonstrates in fact that a tuning fork can be used as sensitive drag force detector. This is best seen if one notices that equation 22b is an effective spring equation with an effective spring constant given by the term in square brackets

\[ k_0 = \eta m_0/(\sqrt{3}Q_0) \]  

The effective spring constant \( k_0 \) is made smaller than the static spring constant by a factor \( \sqrt{3}Q_0 \). By driving the fork on resonance. Since \( Q_0 \) can be made of the order of \( 10^3 \), this clarifies why the arms of a tuning fork which are very stiff in comparison to levers normally used for atomic force microscopy, can be made dynamically softer so to speak. As we will see it shortly, this enhancement of the effective spring constant is only achieved frequency band centered around \( f_0 \) with a band-width \( f_b = \pi \Delta f / \sqrt{3} \). Eq. 22b shows that one can attain a quantitative information about the maximum friction force \( F_{\text{Lo}} \) detected by the tuning fork. Both \( Q_0 \) and \( u_{\text{Lo}} \) are quantities which are quite simple to obtain from independent measurements. 1 The value of \( k_0 \) is also needed in order to determine \( F_{\text{Lo}} \). It can be either calculated using equation 4 or determined directly by measuring the resonance frequency \( f_0 \) of the fork and using equations 15 and 16. Both methods were found to give values of \( k_0 \) consistent with each other within a few percent.

This harmonic oscillator model analysis holds of course for higher harmonic resonances of the arms vibrations. In this case, one needs to replace the subscript 0 by \( n \), the order of the harmonics, in all the above equations. It occurred to us that it might be more desirable to design a tuning fork operating on its higher harmonic frequencies in order to increase the sensitivity of the drag detector. This is best seen using eq.11 in together with equations 20 and 23. For higher harmonics, the effective dynamic spring constant of the arm is

\[ k_n = k_0/n^2 = k_{\text{st}}/\sqrt{n^2/3Q_0} \]  

corresponding for instance to a sensitivity enhancement of a factor 300 over \( k_0 \) should one work at the fifth harmonic. With this in mind, we speculate that such detectors when designed with the proper contact geometry should permit shear force imaging with atomic resolution. Writing eq.24 we have made the assumption that \( \Delta f \) does not depend on the frequency. This approximation is very reasonable since \( \Delta f \) reflects the mechanical losses due to imperfections in the quartz, in the optical fiber and in the cement that holds them together. In the present arrangement, such losses do not depend strongly on the frequency up to the MHz range.

The detection band width \( f_b \) of the present tuning fork force detector is given by solving the equation of motion 13 for transient solutions. This is done by setting switching of the drive force \( F \) at \( t=0 \). The vibration of the arms will relax then freely with a time constant \( \tau \). Such a relaxation time reflects the typical detection band width \( f_b = 1/\tau \). Eq.13 is then a simple linear differential equation with no driving force term, its physical solution is

\[ u_{\text{Lo}}(t) = u_{\text{Lo}}(0) \exp[i(\omega - 2\gamma t)/\omega] \exp[-\gamma t/2] \]
This corresponds to an oscillating function (slightly frequency shifted) with an exponentially decaying envelope with a relaxation corresponding to

$$1/\tau = \gamma/2$$  \hspace{1cm} (26)

Making use of the definition given in eq 21 for $\gamma$, the band width is easily obtained

4. PIEZO-ELECTRIC SIGNAL CONSIDERATIONS

We derive in this section the relation between the amplitude $u_L$ of the tip oscillation and the piezoelectric signal induced by the corresponding deformation of the fork arms. When the arms of the tuning fork bend in their oscillatory motion, a longitudinal tensile stress $\sigma(x,y)$ oriented along $Y$ appears locally at the coordinate $x,y$ of the arms cross section. This stress in turn induces locally a polarization $p(x,y)$ of the piezoelectric material responsible for the piezoelectric signal picked up on the contacts appropriately placed on the tuning fork. The polarization and the stress are related through a local tensor relation $p=\epsilon d\sigma$ where $\epsilon$ is the piezoelectric tensor of the fork material. For sake of simplicity, we will not develop in the present work the full tensor relationship between $p$ and $\sigma$, the interested reader will find a expanded account on this topic in ref.6. We specializing to the case of quartz-like piezo electric material (i.e. Trigonal crystal of class 32) and to the particular case were the axis of fig.1 correspond to the crystal axis X,Y and Z of the fork. In this case, the polarization $p$ is oriented along $X$ perpendicular to the tensile stress and the tensor relation between $p$ and $\sigma$, reduces to a scalar equation

$$p = d_{11}\sigma = -d_{11}\sigma$$

where $\sigma$ is the only nonzero component of $\sigma(x,y)$ (i.e. oriented along $Y$) and $d_{11}$ is the longitudinal piezoelectric modulus. We need now to determine $\sigma$ as a function of $u_L$, the bending of the arm at the tip level. The local expression for Hook's law at coordinate $x,y$ in the arms cross section is $^5$

$$\sigma/E = x \partial^2 u / \partial y^2$$  \hspace{1cm} (28)

We make use of eq.3c for $u(y)$. Eq.28 reduces then to

$$\sigma(x,y) = 3E u_L (L-y) x / L^3$$  \hspace{1cm} (29)

The above equation shows that the longitudinal tensile stress is maximum for $y=0$ which is the plane where the arms are anchored, and for $x=\pm T/2$ which are the outer rim planes of the arms. It is now possible to connect the polarization $p$ to $u_L$

$$p(x,y) = -3d_{11}E u_L (L-y) x / L^3$$  \hspace{1cm} (30)

As expected, the piezo-electric induced local polarization is proportional to the tip amplitude $u_L$. When no external electric field is applied on the contacts pads of the tuning fork, the local electric field is given by $-p/o$. The corresponding local potential per bending unit is

$$V(x,y)/u_L = (3/2)(d_{11}/o) E (L-y)^2 / L^3$$  \hspace{1cm} (31)

A potential drop is obtained between the planes $x=\pm T/2$ and $x=0$. The maximum achievable potential drop is expected between $(y=0, x= T/2)$ and $(y=0, x= -T/2)$

$$V_{max}/u_L = (3/4)(d_{11}/o) E (T/L)^2$$  \hspace{1cm} (32)

For quartz $d_{11}=2.31 \times 10^{12}$ C/N (or equivalently V/m) and $E=7.87 \times 10^{10}$ N/m$^2$. The maximum signal detected on such a fork would be as large as $V_{max}/u_L=15.4 \ (T/L)^2$ in units of volts per nanometers. This figure is three order of magnitudes larger than that measured with a commercial tuning fork for which T=0.6 mm and L=4mm and with contact pads geometry as shown in fig. 1. We speculate that this discrepancy is due to the fact that commercial tuning forks have contact pads designed in order to excite the fundamental mode of oscillation in which both arms oscillate in opposite direction (in a way that would keep the center of mass of the fork fixed in space). In contrast, in the present mode of operation, the dithering excites center of mass oscillation of the tuning fork arms (i.e. arms moving in the same direction, in phase). For a perfect tuning fork with identical arms and with contact pads such as shown in fig.1, the center of mass excitation should give a zero net piezo-electric signal. Because one of the arms is holding the optical fiber tip, the fork is not perfectly symmetric. In this case the arms will oscillate with slightly different amplitudes giving a finite net piezo-electric signal. Its is therefore desirable to redesign the geometry of the contact pads in order to increase the piezo-electric signal. In particular, eq.32 indicates that two extra contact points picking up the piezo-electric signal should be added beside the two contact pads coupling piezoelectrically the two arms of the fork. They should be placed on regions of maximum stress around $(y=0, x= T/2)$ and $(y=0, x= -T/2)$.
5. QUANTITATIVE EVALUATION OF THE SHEAR FORCE:

In this section, we relate the measured piezo-electric signal to the drag force experienced by the tip when it is in shear force interaction proximity with the sample. In feedback operation mode, the quartz tuning fork is mechanically excited at its fundamental resonant frequency. When the tip is far away from the sample surface, \( u_{t0} \) is maximum and the corresponding piezo-electric signal is also maximum. As the tip is approached, the amplitude of the signal reduces as \( u_{t0} \) reduces. If the driving force is maintained constant during the tip sample approach, a reduction of the tip amplitude corresponds to a proportional reduction of \( Q_0 \) as indicated by eq. 22a and \( u_{t0}/Q_0 \) remains therefore constant. This, as seen from eq. 22b, implies that the drag force \( F_{DP0} \) detected by the tuning fork is unaffected by the tip sample interaction. It is important to realize that the tuning fork react to the tip sample approach in a way that keeps the total drag force constant. A piezo-electric tuning fork tip-sample distance control system operates at total constant drag force. This observation indicates in appearance that such a device fails to inform us about the tip-sample friction forces. This is in fact not the case. As we mentioned earlier, the total drag force acting on the arm of the tuning fork is a composite drag force which is the sum of a parasitic drag force \( F_p \) internal to the device and of the tip-sample drag force \( F_s \). When the tip is away from the sample surface, the device detects only the nominal parasitic drag force \( F_{DP0}=F_{DP0}=F \). When the tip is in interaction range, the drag force acting on the fork is \( F_p+F_s=F \). While the total detected drag force is constant, the various contributions making it are changing when the tip approaches the sample surface. This leads us then to write:

\[
F_s=F_{DP0}(1-F_p/F_{DP0})
\]  

(33)

where \( F_{DP0}=F_{DP0} \) is given by eq. 22b. The nature of the parasitic drag force obviously does not depend on the tip-sample distance. Consequently \( F_p \) differs from \( F_{DP0} \) only due to the fact that the tip amplitude reduced upon approach. We can therefore write that the amplitude of the parasitic drag force changes proportionally to the piezo-electric signal:

\[
F_p/F_{DP0}=V/V_0
\]  

(34)

where \( V_0 \) and \( V \) are the detected piezo electric signals when the tip is away from and in interaction range with the sample respectively. The shear force picked up by the tip is then

\[
F_s=(1-V/V_0)[k_{sat}/(\sqrt{3}Q_0)] u_{t0}
\]  

(35)

Where \( Q_0 \) is the quality factor of the fork and \( u_{t0} \) is the corresponding oscillation amplitude when the tip is far from the sample. Since \( Q_0 \), \( u_{t0} \), and \( k_{sat} \) are quantities that are easily obtained, the above equation shows that the tip-sample interaction shear force can be evaluated quantitatively. We note that the knowledge of the strength driving force as well as the piezo-electric parameters of the fork are not explicitly required in order to obtain the tip-sample interaction force.

6. NOISE LIMIT CONSIDERATIONS

As we have seen it in the previous sections, the tuning fork can be modeled as a harmonic oscillator with a one dimensional degree of freedom. The theoretical noise limit is in principle derived from a statistical mechanics model of a harmonic oscillator. We will present here the most simplified noise analysis based on the equipartition theorem. This analysis is in fact correct when the measurement band width is only self limited by the intrinsic frequency band width \( f_0 \) of the tuning fork. This situation corresponds to fastest, and therefore the desirable, tip-sample distance control mode of operation. The equipartition theorem dictates that the average harmonic oscillator energy equates \( k_B T_e/2 \) the average thermal energy at temperature \( T_e \):

\[
\frac{1}{2} k_{sat} u_{t0}^2 = \frac{1}{2} k_B T_e
\]  

(36)

where \( u_{t0}^2 \) is the average amplitude of the fork arm thermal fluctuations and \( T_e \) is the temperature of the fork. The noise floor in the self limited band width \( f_0 \) around the fork resonance frequency is then given by

\[
<\Delta u_{t0}^2>^{1/2} = \frac{k_B T_e}{k_{sat}}^{1/2}
\]  

(37)

For most practical tuning fork applications \( k_{sat} \) ranges between 10^3 and 10^5 N/m, making \( <\Delta u_{t0}^2>^{1/2} \) of ranging between 2x10^{-13} m and 2x10^{-12} m at room temperatures. The equivalent noise drag force detected by the fork is:

\[
<\Delta F_{DP0}>^{1/2} = [k_{sat} k_B T_e]^{1/2}/(\sqrt{3}Q_0)
\]  

(38)

For, \( k_{sat}=26000 \) N/m^2 and \( Q_0=1000 \), the equivalent drag-force noise is 6pN at \( T=300K \) and 0.7pN at \( T=4.2K \). We have observed that apertures at the apex of aluminum coated tapered optical fiber tips are destroyed when shear force exceeds 3 to 10nN. This indicates that the useful piezo-electric signal to noise ratio can be made of the order 10^5 in a single band width \( f_0 \). In the results presented in ref.1, we measured an equivalent drag force noise at 300K of about 20pN. In this case the noise was due to our amplification electronics. An improvement of a factor three can be in principle acheived.

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7. A KNOWN DRAG FORCE: THE HYDRODYNAMIC VISCOUS DRAG ON A MOVING CYLINDER:

We have performed hydrodynamic measurements in order to evaluate in an independent way the magnitude of the drag forces detected by the piezo-electric tuning fork. The idea is to measure the viscous drag experienced by a flat cleaved optical fiber when it is oscillating in a liquid. The configuration of the hydrodynamic measurements is shown schematically in fig.5. In this geometry, a flat cleaved 125 μm diameter cylindrical optical fiber is glued along on the tuning fork prongs. The cylindrical fiber is protruding 1mm outside the prong, as indicated in fig.*.

![Quartz Tuning Fork, Optical fiber, Direction of oscillation, Oil, Cleaved optical fiber (125mm μm)](image)

**fig.5** Schematic of the hydrodynamic measurement geometry. The flat cleaved 125 μm diameter cylindrical optical fiber is dipped into silicon oil. The viscous drag experienced by the fiber manifests itself in a reduction of both the fork oscillation amplitude and Q.

The protruding cylindrical fiber element is dipped with a depth d into an oil with known viscosity. We have chosen this geometry because the viscous drag of a cylinder moving is known and has an analytical expression for small oscillation amplitude (i.e. for small Reynolds numbers). In this mode of operation the tuning fork is still vibrated on its mechanical resonance with the dither tube. At constant dither drive, the deeper is the fiber in oil (i.e. the larger is d), the larger is the viscous drag experienced by the fiber. As a result of this, as d increases, both the Q factor and the prongs oscillation amplitude reduce. We have measured the frequency dependence of the fork piezo-electric signal for different depths d of the fiber in oil. The values of the resonance position of the peak piezo-signal, its FWHM and its amplitude are shown in fig.6 as a function of d. It is of relevant importance to say that the measurement were performed for decreasing values of d. As the fiber is pulled out of the oil, the piezo-electric signal amplitude increases. At d=0 the amplitude increases abruptly by about a factor 2 as seen in the top panel of fig.6. At the same time the FWHM decrease abruptly by a factor 2 as seen in the lower panel of fig.6. This abrupt transition corresponds to the situation in which the oil meniscus surrounding the fiber brake away setting the fiber free from the oil viscous drag. The middle panel of fig.6 shows the frequency shift of the resonance. For small d, there is no measurable shift in the resonance position, while in the same time the amplitude of the signal as well as its FWHM decrease by more than a factor 2. This shows very convincingly that the nature of the forces picked up by the fiber are purely lossy in agreement with the equation of motion of the fork prongs. As d increases the resonance position is shifted toward lower frequency. This frequency shift is fitted with a quadratic dependence in d as shown in the middle panel of fig.6.

![Graphs showing Amplitude (µV), Δf (Hz), FWHM (Hz)](image)

**Fig.6:** Piezo-electric resonance signal characteristic as a function of the depth d of the fiber dipped in silicon oil. For d=0, the resonance is centered at 33.1 kHz. **Top panel:** The piezo-electric signal amplitude is proportional to the fork’s prong oscillation amplitude with a proportionality constant of 27μV/nm. The jump in signal at d=0 corresponds to the separation between the fiber and the oil meniscus. **Middle panel:** frequency shift of the resonance as a function of d. The full line is the best quadratic fit to the data. Note that there is no measurable frequency shift at d=0 when the fiber breaks away from the oil. **Lower panel,** the corresponding full width at half maximum of the piezo-electric resonance (the full line is a guide to the eye).
The functional dependence of Q with d is obtained by using the measured values of the resonance position divided by the FWHM shown in fig.6. Since this way, Q and the resonance amplitude are both obtained independently, we can check the linearity of Q with x predicted in our simple effective mass harmonic oscillator model. This linear dependence is demonstrated in fig.7, where Q is plotted against the signal amplitude. In this representation d is now a hidden variable. This shows that, as d increases, x but also Q decrease in the same proportions. Since the total drag force \( F_D \) sensed by the tuning fork is given by \( F_D = k \nu / (Q \sqrt{3}) \), we conclude that \( F_D \) remains constant and is independent on d.

\[
F_{\text{fcl}} = 8\pi \mu d |\partial u_1 / \partial t| / \left[ 1 - \ln(1.781 \rho \Phi \partial u_1 / \partial t / \mu) \right] \tag{39}
\]

The motion of the fiber is however not uniform but oscillatory. As a result of this, the oil displaced by the dipping fiber is accelerated back and forth. This causes an inertial drag acting on the fiber which simply given by the Newton equation for the displaced mass of oil \( m_{\text{oil}} \):

\[
F_{\text{int}} = m_{\text{oil}} |\partial^2 u_1 / \partial t^2| = \left( \pi / 8 \right) \Phi^2 d \omega^2 u_1 \tag{40}
\]

The estimated calculated hydrodynamic drag experienced by the tip is then given by \( F_{\text{fcl}} + F_{\text{int}} \). In fig.8 we have calculated this force normalized to a unit of fiber oscillation amplitude. The agreement between this calculation and the total drag experienced by the fork as experimentally determined using \( F_D \nu_0 = k / (Q \sqrt{3}) \) is generally not satisfactory. The model calculation is oversimplified since it does not account for the oil meniscus forming around the fiber. For larger d however, the surface tension contribution compared to the bulk contribution to the viscous drag should diminish. We expect then a better agreement between the model and the measured data for larger d. This is what is observed in fig.8. Apart for a constant shift, both the calculated and measured \( F_D \nu_0 \) have a similar dependence on d. This constant shift is due in part to the drag force internal to the tuning fork. Given the fact that no adjustable parameters were used in the calculation we find the calculated result surprisingly close to the measured one. The slope of the calculated drag shown in fig.8 was found to depend in a sensitive way on the viscosity of the liquid. As a consequence a practical application of the tuning force used as drag detector would be to use it as a viscometer.

![Graph](image)

**Fig.7:** The Q values obtained from the data of fig.6 are plotted against corresponding the fiber amplitude. The depth d of the fiber dipping in oil is eliminated between the two quantities. The linearity between Q and the fiber amplitude is expected from the simple effective mass harmonic oscillator model.

It is now interesting to now to compare the total drag force experienced by the fork to the calculated viscous drag experienced by the fiber oscillating in the oil. The viscous drag acting on a moving cylinder inside a fluid is analytically known in the limit of small velocities. This limit known as that of small Reynolds number regime is characterized by the dimension less Reynolds number \( R = \rho \Phi \omega / \mu \) where \( \rho = 960 \text{ Kg/m}^3 \) is the density of the silicon oil we used, \( \mu = 2.05 \text{ Pa.s} \) is its viscosity \( \Phi = 125 \mu \text{m} \) is the cleaved fiber diameter and \( |\partial u_1 / \partial t| = \omega \nu_1 \) is its velocity. For 'large' \( \nu_1 \) amplitudes of the order of 10 nm, we estimate \( R = 1.2 \times 10^4 \ll 1 \) showing that the small Reynolds regime approximation is fully justified. The hydrodynamic drag force acting on the fiber is given by Lamb theory:

\[
F_{\text{cl}} = \frac{8\pi \mu d |\partial u_1 / \partial t|}{1 - \ln(1.781 \rho \Phi \partial u_1 / \partial t / \mu)} \tag{39}
\]

The motion of the fiber is however not uniform but oscillatory. As a result of this, the oil displaced by the dipping fiber is accelerated back and forth. This causes an inertial drag acting on the fiber which simply given by the Newton equation for the displaced mass of oil \( m_{\text{oil}} \):

\[
F_{\text{int}} = m_{\text{oil}} |\partial^2 u_1 / \partial t^2| = \left( \pi / 8 \right) \Phi^2 d \omega^2 u_1 \tag{40}
\]

The estimated calculated hydrodynamic drag experienced by the tip is then given by \( F_{\text{fcl}} + F_{\text{int}} \). In fig.8 we have calculated this force normalized to a unit of fiber oscillation amplitude. The agreement between this calculation and the total drag experienced by the fork as experimentally determined using \( F_D \nu_0 = k / (Q \sqrt{3}) \) is generally not satisfactory. The model calculation is oversimplified since it does not account for the oil meniscus forming around the fiber. For larger d however, the surface tension contribution compared to the bulk contribution to the viscous drag should diminish. We expect then a better agreement between the model and the measured data for larger d. This is what is observed in fig.8. Apart for a constant shift, both the calculated and measured \( F_D \nu_0 \) have a similar dependence on d. This constant shift is due in part to the drag force internal to the tuning fork. Given the fact that no adjustable parameters were used in the calculation we find the calculated result surprisingly close to the measured one. The slope of the calculated drag shown in fig.8 was found to depend in a sensitive way on the viscosity of the liquid. As a consequence a practical application of the tuning force used as drag detector would be to use it as a viscometer.

![Graph](image)

**Fig.8:** Drag force per unit of prongs displacement as a function of the depth of the fiber oscillating in oil. (A) is obtained by using equations 23 with the measured Q's, the line through the data is a guide to the eye. The jump in the data at d=0 is due to the abrupt separation between the fiber and the oil surface as d is decreased from 0'0 to 0'. (B) is the calculated viscous drag force experienced by the fiber using the idealized model described in the text.
8: FRICTION FORCE AND NSOM IMAGES:

In this section we present a number of representative examples showing that the friction force detector operates to our satisfaction in order to control the tip-sample distance. The quality of the following friction force images are however rather poor in comparison to the intrinsic noise limitation of the tuning-fork friction force detector. The reason for this, is the rather poor thermal stability of our microscope design. We are currently building a microscope expected to reach the intrinsic friction resolution limit due to the room temperature brownian motion of the tuning fork arms. All the following images were taken in non contact mode (with typically 5nm tip sample separation. The details concerning the images are described in the captions and are ordered by decreasing image size and increasing sensitivity in the vertical range.

Fig.9A and Fig.9B are the simultaneous friction force image and the near field optical image of red blood cells. The image size is about 15x15μm. In this set up, the blood cell smear was freshly prepared (i.e. not dried) on a thin cover glass which was directly glued above the 4x4mm photosensitive region of a commercial Si PIN photodetector. This way the near field optical signal is collected in transmission with in a very efficient and easy manner. The vertical scan range between the darkest (i.e. deeper) and brightest (i.e. higher) regions in the friction image was about 2μm. The optical contrast between the darker and lighter region of the near field image is about 12%.

Fig.10A: Three dimensional plot of a constant friction force (of the order of 2 nN) image taken on a GaAs sample with a holographically patterned 100 nm thick photoresist. The resulting sample topography shows structures periodic along X (700nm period) and 80nm high. The image was taken over 1.8x1.2μm and was acquired in 200 sec with an average of 5msec./pixel. The tuning-fork resonant frequency was 100 kHz. Fig.10B is a constant friction line scan taken across X taken in the middle region of fig.10A.
Fig. 11A is the friction force image taken over a GaAs crystal sample on which two metal interlocked comb gates were fabricated using electron beam lithography. The thickness of the metal fingers is 20nm. The image size is 1.5x1.5μm. The image shows the region where both gate combs start to overlap. The structures running vertically across the image corresponds to the gate fingers. The periodic separation between overlapping fingers is 232nm. Fig. 11A is corrected for slow (thermal) drifts by performing a spatial numerical derivative. This transformation increases the noise. Fig. 11B shows two unprocessed line scans taken across the gate finger over a region where both gates overlap (lower panel), and where they do not overlap (upper panel). The solid line in the lower panel is a best fit to a cos of curve with period 232 nm.

Fig. 12A

Fig. 12B

Fig. 12C

Fig.12A Shows unprocessed friction force image of the surface of a GaAs crystal with growth defects. The image size is 1μm x 1μm with a resolution of 200x200 pixels. The integration time was 16 msec/pixel, the total acquisition time was 642 sec. This image is of poor quality because of slow thermal drifts in our poorly designed microscope. Such thermal instability are slow enough that it does not affect appreciably the scan over a line scan. Fig. 12B shows a line scan indicated by the marker in fig.12A. The typical height noise in this measurement is about δZ=0.2 nm. In fig.12C the long term thermal drift are eliminated by plotting the spatial derivative of fig.12A with respect to X, this digital transformation increases high frequency noise.
9. FUTURE DEVELOPMENTS: USING THE TUNING FORK WITHOUT AN EXTERNAL DITHER

Up to now we have considered the case for which the tuning fork friction force detector is mechanically excited on its resonance by making use of an external piezoelectric vibrator (i.e., external dither piezo). We present in this section a way to use the fork without external mechanical excitation. The tuning fork itself can be used as its own vibrator by using the converse piezoelectric effect. When the contact pads A and B of fig.1 are connected to an external low voltage oscillator, the arms of the tuning fork are set into oscillatory motion. The fork can be driven this way with a drive voltage \( U_{EXC} \) on its resonance frequency. The signal to be monitored in this mode of operation, is the corresponding a.c. current flowing through the tuning fork. At frequencies away from resonance conditions, the tuning fork and the contact pads is nothing more than a capacitor \( C_p \) (typically a few pF for commercial 33kHz quartz forks). The resulting current is \( I_{CAPA}=2\pi f C_p U_{EXC} \) increasing linearly with the frequency. When the fork is driven on its resonance frequency, the current peaks to a value \( I_{MAX} \), which intensity depends on the drag force experienced by the fork. This peak current value is the useful signal to be used for tip-sample distance control. In contrast with the case of external mechanical excitation of the fork, in this mode of operation, the useful part of the signal is interfering with the capacitive parasitic background signal \( I_{CAPA} \). Such a behavior of the current is given by equation 41 which is derived using an analysis very similar to that presented in section 4. Writing that the current density \( j \) and the electric displacement \( D \) field in the fork satisfies the low frequency condition \( j=\partial D/\partial t \approx 0 \) we find that the current amplitude through the fork is given by

\[
I = 2\pi f C_p | j(\delta V/\delta t) E (T/L)^2 u_L + U_{EXC} | \quad (41)
\]

Where \( \eta \) is a constant of the order of unity indicative of the geometry of the contact pads and \( u_L \) is the tip amplitude given in equation 25. The first term in the brackets is the component that contains the shear force, its amplitude is large only around resonant conditions. In contrast, the second term, which is the drive potential, is responsible for the capacitive background signal. It is worth mentioning that one cannot reduce the drive voltage \( U_{EXC} \) in order to reduce the background signal. This is because \( u_L \) is also proportional to \( U_{EXC} \). Rather than relying on eq.41 for the current, we have instead measured it for a typical experimental condition. A measurement of the current flowing through a quartz tuning fork mounted with an optical fiber tip is shown in fig.13. We have carried such type of measurements for a wide range of \( Q_0 \), and for the three different quartz tuning forks presented in table 1, and we found that \( I_{MAX}/I_{CAPA} \approx Q_0 f_0 = \alpha \Delta f \) where \( \alpha \approx 110 \) is a constant depending on the nature of the fork material.

![Graph](image)

Fig 13. The current flowing through the piezoelectric tuning fork is measured under constant voltage excitation \( U_{EXC} \) (i.e., 1mV peak to peak) as a function of the frequency of this excitation. This voltage excitation is applied on the contact pads of the tuning fork. The points are measured, the full line is calculated using equation 42. Away from resonance condition the tuning fork acts as a capacitor. The current \( I_{EXC} \) flowing through it, is indicated by the horizontal dashed line. On resonance, the vibration amplitude of the tuning fork is about 0.4nm peak to peak for this particular measurement. In this mode of operation tip-sample distance control can be achieved without external dither.

The ratio \( I_{MAX}/I_{CAPA} \approx 1 \) represents the contrast ratio of the useful signal to the parasitic \( I_{CAPA} \). For \( Q_0=2176 \), \( f_0=33kHz \), \( L=4mm \), \( T=0.66mm \) \( \Sigma=0.4mm \), the contrast ratio is measured to be \( I_{MAX}/I_{CAPA} \approx 6.40 \). The fork is driven with a peak to peak amplitude of \( U_{EXC}=1mV \). Under such conditions, and far from the sample, the resonant tip vibration amplitude is roughly 0.5nm. The corresponding measured currents was \( I_{MAX}=1.1nA \) at \( f_0 \) and \( I_{CAPA}=0.149nA \) at 30kHz. As seen in fig.13, the shape of the measured current peak is an asymmetric function of the frequency. The reason for this is that the phase of the piezoelectric current contribution changes by 180 degrees as the frequency is swept past the resonance. On the other hand the capacitive current is of constant phase. As a result, both the piezo-electric and the capacitive contribution to the current interfere. It is simple to simulate such a behavior by writing a phenomenological equation similar to eq.41:

\[
I/I_{CAPA} = | \Delta \{ \gamma \omega/(\omega_0^2-\omega^2+i\gamma \omega) \} +1 | \quad (42)
\]

where the first term in the right term is proportional to the fork's arm amplitude with a proportionality constant given by \( \Delta^2 = (I_{MAX}/I_{CAPA})^2 \approx 1 \). The constant second term represents the contribution of the capacitive current leakage which interferes with the first term. The best fit to the data of fig.13 is given for \( Q_0 \), \( f_0 \) and \( I_{MAX}/I_{CAPA} \) indicated in the
figure. In this new mode of operation since no external
vibrator is needed, the tip-sample friction force detector is
reduced to the tuning fork itself, making it possible to
have extremely compact (i.e. couple of cm to sub-cm size)
near field optical scanning microscope stand alone heads.
Preliminary friction force measurements were obtained and
have demonstrated that a satisfactory tip-sample distance
control is made possible in this mode of operation. It is still
desirable to perform a careful calibration of the amplitude
of the tip oscillation with the measured current $I_{\text{MAX}}$ under
a given $U_{\text{EXC}}$. Such a calibration measurement is under
way and is expected to be independent of $Q$ for all forks of
same size and contact pad geometry.

10. CONCLUSIONS:

We have primarily developed the piezo-electric tuning
fork tip-sample distance control with the intent to operate
the NSOM at low temperatures or/and at high magnetic
fields. Satisfactory tip-sample distance control was obtained
when the microscope system was immersed in 1.5 K
superfluid helium. At room temperatures, the tip-sample
distance control was also found to operate in the 1 tesla
field of a permanent magnet. The weak point of the quartz
fork detection scheme is that the resonance frequency $f_0$
and $Q$ depends on the temperature of the quartz. Such dependence does not appear to be critical in
normal room temperature operation condition since have
obtained NSOM images recorded on time scale ranging
between 15 minutes to 2 hours with out loosing the tip-
sample distance control. At low temperatures the $Q$
factor can increase by as much as an order of magnitude. Low
temperature measurements should be performed with
stabilized temperatures. We have also performed
preliminary measurements in a magnetic field of 1T without
noticing any change in the piezo-electric signal. Ramping
the magnetic field creates however eddy currents in the
contact pads. For this reason tip-sample distance control
should be done at constant magnetic fields.

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