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Operation characteristics of piezoelectric quartz tuning forks in high magnetic fields at liquid helium temperatures

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Piezoelectric quartz tuning forks are investigated for use as force sensors in dynamic mode scanning probe microscopy at temperatures down to 1.5 K and in magnetic fields up to 8 T. The mechanical properties of the forks are extracted from the frequency dependent admittance and simultaneous interferometric measurements. The performance of the forks in a cryogenic environment is investigated. Force-distance studies performed with these sensors at low temperatures are presented.

I. INTRODUCTION

Piezoelectric quartz tuning forks were introduced into scanning probe microscopy by Günther, Fischer, and Dransfeld[1] for use in scanning near field acoustic microscopy and later by Karrai and Grober[2] as a distance control for a scanning near field optical microscope (SNOM). Several other implementations of tuning forks have been reported, e.g., in SNOMs,[3–6] scanning force microscopes (SFMs),[7,8] magnetic force microscopes[9] and in the acoustic near field microscope.[10] Operation in a cryogenic environment was reported by Karrai and Grober in their pioneering work.[2] To our knowledge operation characteristics at temperatures below 10 K have not been reported to date. In this article we present results on piezoelectric tuning fork sensors in our low temperature SFM which operates in the sample space of a ^{4}He cryostat.[11]

In our studies we utilized commercially available tuning forks [see the inset of Fig. 1(a)] which are usually employed in watches with a standard frequency of 2^{15} Hz. These forks are fabricated from wafers of α quartz with the optical axis oriented approximately normal to the wafer plane.

The tuning fork can either be mechanically driven by an additional piezo element or electrically excited through the tuning fork electrodes.[12] Similar to the method used in Ref. 7 we drive the oscillation electrically by applying an ac voltage of typically $U=0.01-10\,\mathrm{mV}$ to the tuning fork contacts. For investigation of the tuning fork behavior we measure the complex admittance of the fork with a two-channel lock-in amplifier. When employed as the sensor for dynamic force measurements the tuning fork is part of a phase-locked loop that is described in Ref. 11.

II. CALIBRATION OF THE OSCILLATION AMPLITUDE

Figure 1(b) shows typical resonance in the admittance of a tuning fork measured at room temperature at a pressure of 6×10^{-7} mbar. The admittance exhibits asymmetric resonance at 32 768 Hz and a sharp minimum about 30 Hz above this resonance. The current through the fork consists of two parts:[12] I_p is the current created by the mechanical (harmonic) oscillation of the fork arms through the piezoelectric effect of the quartz and I_0 is the capacitive current through the fork. The behavior of the admittance can therefore be modeled with the equivalent circuit shown in the inset of Fig. 1(b).

The LRC series resonator with a resonance frequency $f_0=1/(2\pi\sqrt{LC})$ around 2^{15} Hz and a quality factor $Q=\sqrt{L/(CR^2)}$ which is typically of the order of 10^4 allows the current I_p to pass. Using a mechanical model one can relate L, R and C to the effective mass of one arm m, the damping constant γ, the spring constant k and the driving force αU via $L=m/(2\alpha^2)$, $C=2\alpha^2/k$, $R=m\gamma/(2\alpha^2)$. The capacitance C_0 is mainly determined by the geometrical arrangement of the contacts on the crystal, the dielectric properties of the quartz and by cable capacitances. The fit to the measured admittance in Fig. 1(a) (which could not be distinguished in the plot from the measured curve) leads to $C_0=1.2129\,\mathrm{pF}$, $C=2.9\,fF$, $L=8.1\times10^3\,\mathrm{H}$, $R=27.1\,\mathrm{k}\Omega$, $f_0=32\,765.58\,\mathrm{Hz}$, and $Q=61\,730$.

In addition to the electrical resonance we measured the mechanical resonance amplitude x of one of the tuning fork arms [see Fig. 1(a)] utilizing the interferometer setup usually used for optical cantilever deflection detection in a scanning force microscope.[13] From a combination of both measurements [Figs. 1(a) and 1(b)] and using the relation $I_p=4\pi f\alpha x$,[12] we determined the effective mass $m=0.332\,\mathrm{mg}$, the quality factor $Q=61\,734$, the spring constant $k=14\,066.4\,\mathrm{N/m}$, and the piezoelectric coupling constant $\alpha=4.26\,\mu\mathrm{C/m}$. The effective mass calculated from the density of quartz and the dimensions of a tuning fork arm turns out to be 0.36 mg, in good agreement with our measured value. A linear relation between the driving voltage and the oscillation amplitude was found in the interferometer measurement down to amplitudes of 1 nm as well as in large-amplitude measurements performed under an optical microscope up to amplitudes of about 100 μm.

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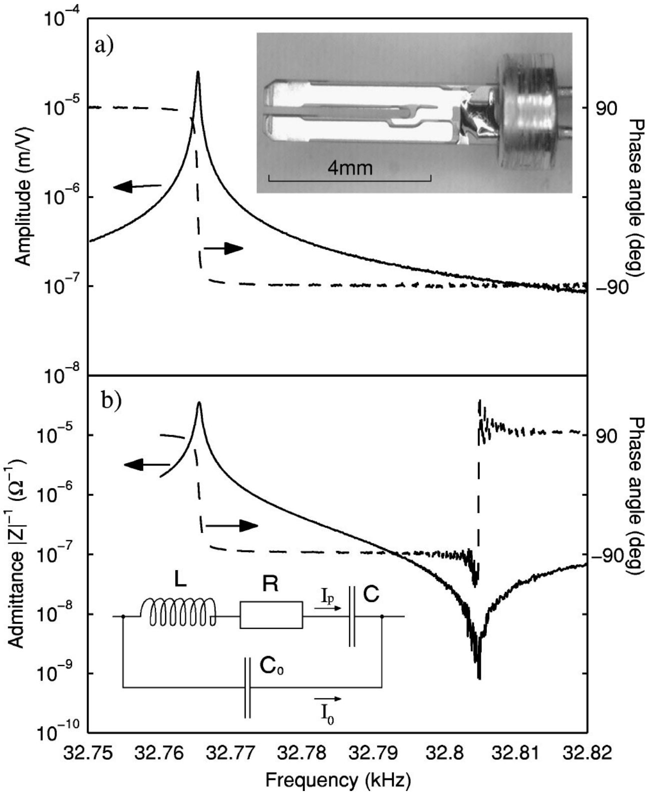

FIG. 1. (a) Mechanical resonance measured at room temperature at a pressure of 6×10^{-7} mbar with an optical interferometer. Inset: Image of the tuning fork. (b) Electrical tuning fork resonance measured simultaneously. Inset: Equivalent circuit for the piezoelectric quartz tuning fork resonator. The solid and dashed lines are the respective amplitude and phase.

III. ATTACHING A TIP TO THE TUNING FORK

For use in our SFM we remove the tuning forks from their casing and glue a thin metallic wire (10–50 μm diam) in the direction of oscillatory motion to the end of one prong. The wire is then etched electrochemically to form a sharp tip. If the wire is electrically connected to one of the tuning fork contacts its length is about 500 μm. In cases where we connect the wire to a separate contact pad the wire can be up to 3 mm long. The additional weight Δm fixed to the tuning fork arm is in the range of 1.5–50 μg. In order to obtain the most sensitive force gradient detection it is important to keep the relative mass increase as small as possible. After this modification the resonance of the tuning forks is always shifted to lower frequency, in most cases less than 100 Hz, and typical quality factors $Q=10\,000$ under ambient conditions are reached.

IV. LOW-TEMPERATURE OPERATION

In our SFM we operate the tuning forks in the gas flow of a variable temperature ^{4}He cryostat. As an alternative the sample space can be flooded with liquid He and the microscope is then operated either in normal fluid ^{4}He or in a mixed normal-superfluid phase (below 2.2 K).[14] Table I shows tuning fork resonance characteristics obtained under these different conditions. Compared to operation in gas the resonance frequency of the fork is shifted by more than 500 Hz to lower frequencies in the normal fluid liquid. At the same time the Q value of the resonator decreases due to the significantly increased friction in the liquid. At temperatures below 2.2 K the quality factor increases again as a result of the formation of superfluid ^{4}He which tends to suppress friction effects. An explanation for the reduction of the resonance frequency in the superfluid has not been found. These measurements demonstrate the robustness of the tuning fork properties when external conditions are changed dramatically. With a conventional cantilever for scanning force microscopy it is difficult to achieve Q values of this order in liquid ^{4}He. However, even with the tuning forks, operation in liquid He is cumbersome because the resonance frequency fluctuates greatly. During scanning the tuning fork is typically operated at a constant frequency shift of 100 mHz and deviations of more than a few mHz induced by the environment cannot be intolerated.

TABLE I. The resonance characteristics of the tuning fork in different helium phases.

	T (K)	f_0 (Hz)	Q
Helium gas ($p<1$ mbar)	≈5	32 634	22 665
Liquid helium	4.2	32 110	2 153
Suprafluid helium	1.56	32 056	7 583

The temperature coefficient of the resonance frequency below 5 K was determined to be 260 mHz/K at a constant pressure of 10 mbar (see Fig. 2). The pressure coefficient was 50 mHz/mbar at 5 K. This means that frequency shifts of the order of 10 mHz are produced by temperature instabilities of about 50 mK or pressure instabilities of about 0.2

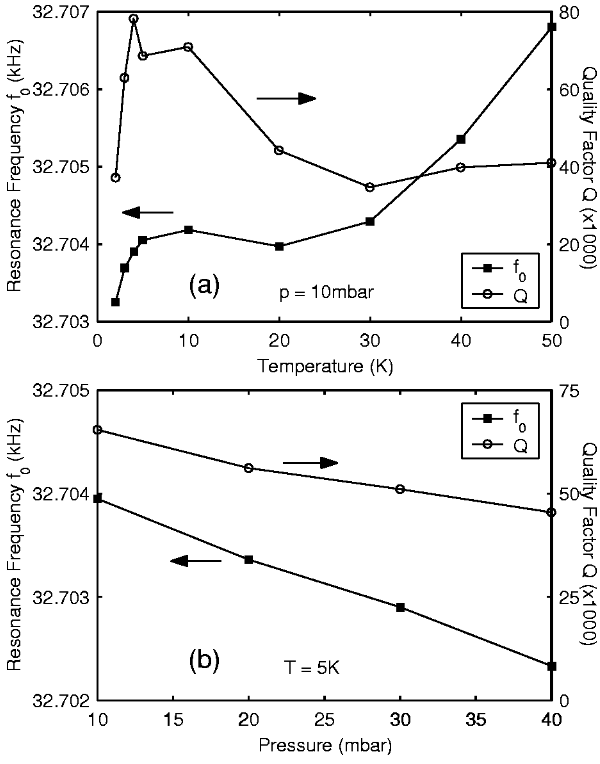

FIG. 2. Measured resonance frequency and Q value (a) vs temperature at a constant pressure of 10 mbar and (b) vs pressure at a constant temperature of 5 K.

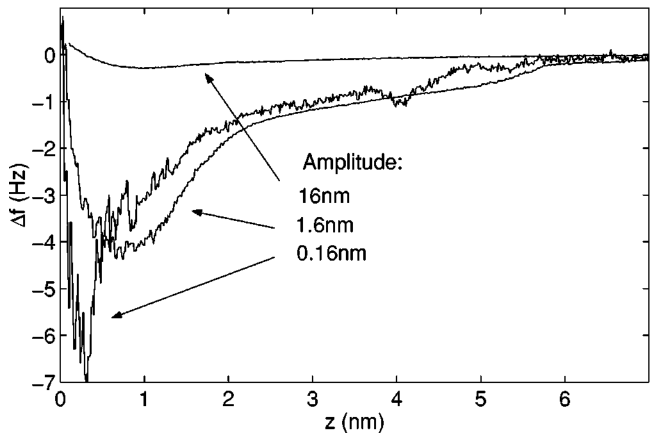

FIG. 3. Measured frequency shift as a function of tip-sample separation for different oscillation amplitudes ($T = 2.5$ K).

mbar. In addition, we measured the dependence of the resonance frequency on an external magnetic field in the range of 0–8 T. The frequency shift detected was smaller than 100 mHz.

V. TIP-SAMPLE INTERACTIONS

In order to demonstrate the power of the piezoelectric tuning fork sensing of tip-sample interactions[15] we show in Fig. 3 a set of measurements of the frequency shift versus distance of the tip to the Au surface measured at 2.5 K and zero tip-sample voltage. From the quality of topographical images taken with this tip just before these measurements we deduce a tip radius of several hundred nanometers.

The oscillaton amplitude was varied by a factor of 10 from one curve to the next, starting from a sub-nm value. A larger oscillation amplitude averages over a larger z range in the repulsive as well as in the attractive region of the interaction such that the frequency shift at a given distance decreases with increasing amplitude. Such behavior was quantitatively described by Giessibl[16] for a attractive van der Waals potential of the form $V \propto z^{-n}$, where n is a positive integer.

VI. DISCUSSION OF THE LOW-TEMPERATURE PERFORMANCE

The typical values of the spring constants of tuning forks are much higher than those of conventional SFM cantilevers. This has several implications for their use in a dynamic mode SFM: first, a given force gradient leads to a smaller shift of the resonance frequency than in conventional cantilevers, since typically $\delta f / f_0 \propto k^{-1}$, no matter whether small or large oscillation amplitudes are used.[16] Care has to be taken in the design of the control electronics to make sure that frequency shifts of at least 10 mHz can be measured to compensate for this disadvantage. Second, there is no danger of the tip snapping into contact with the sample, since the condition $k > \partial F / \partial z$ is met for all tip-sample spacings. This makes the tuning fork an ideal tool to investigate tip-sample interactions as a function of distance. And, last but not least, the high spring constant makes tuning forks ideal for specific applications, e.g., for nanolithography in the noncontact mode[17] or as carriers for all kinds of scanning nanosensors which may be harder to implement on conventional SFM cantilevers. These issues will be discussed in future publications.

The Q values obtained with our tuning forks at pressures around 1 mbar are generally of the same order as the best cantilevers when operated under ultrahigh vacuum (UHV) conditions. The robustness of Q against pressure changes is also significantly higher than that of conventional cantilevers. Compared to piezoresistive cantilevers which tend to heat systems at low temperatures with powers in the 1 mW range tuning forks do not produce any significant amount of heating power and are therefore ideal for future applications in ^{3}He systems or dilution refrigerators.

VII. DISCUSSION

In conlusion, we have demonstrated the operation of piezoelectric tuning forks as sensors for dynamic mode scanning force microscopy at cryogenic temperatures and discussed their performance. The robustness of this sensor allows one to achieve very high quality factors even under the otherwise problematic conditions of non-UHV environments. The force gradient detection method is well suited for force distance studies.

ACKNOWLEDGMENT

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