Interface circuits for quartz crystal sensors in scanning probe microscopy applications

Johann Jersch, a) Tobias Maletzky, b) and Harald Fuchs c)

Physikalisches Institut, Universität Münster, Wilhelm-Klemm-Strasse 10, Münster D48149, Germany

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Complementary to industrial cantilever based force sensors in scanning probe microscopy (SPM), symmetrical quartz crystal resonators (QCRs), e.g., tuning fork, trident tuning fork, and needle quartz sensors, are of great interest. A self-excitation scheme with QCR is particularly promising and allows the development of cheap SPM heads with excellent characteristics. We have developed a high performance electronic interface based on an amplitude controlled oscillator and a phase-locked loop frequency demodulator applicable for QCR with frequencies from 10 up to 10 MHz. The oscillation amplitude of the sensing tip can be set from thermal noise level up to amplitudes of a tenth of nanometers. The device is small, cheap, and highly sensitive in amplitude and frequency measurements. Important features of the design are grounded QCR, parasitic capacity compensation, bridge schematic, and high temperature stability. Characteristic experimental data of the device and its operation in combination with a commercial SPM and a homemade scanning near-field optical microscope are reported. By using the 1 MHz needle quartz resonator with a standard atomic force microscope tip attached, atomic scale resolution in ambient conditions is achieved. Furthermore, reproducible measurements on very soft materials (Langmuir-Blodgett layers) with a very stiff needle quartz (∼400 000 N/m) are possible. © 2006 American Institute of Physics. [DOI: 10.1063/1.2238467]

INTRODUCTION

Quartz crystal resonators (QCRs) belong to the most frequently used sensors in engineering because of their low internal energy dissipation, high temperature as well as mechanical and chemical stabilities, and relatively high piezoconstants. Recently, various QCR probe systems such as tuning fork (TF), trident TF, and needle quartz were introduced as a platform for an attached force-sensing tip in different scanning probe microscopy (SPM) techniques such as acoustic, force, near-field optical, and magnetic force microscopies (see review article5). A high quality factor, even in air, and high spring constants are prerequisites for resolving short range forces and stable operation in force microscopy. Furthermore, very low energy dissipation at low excitation amplitudes, the possibility to maintain a subnanometer tip-sample gap without repulsive mechanical contact (snap in) and low dielectric losses even at high frequencies are crucial factors in specific cases, e.g., in scanning microwave or capacitance microscopy.

There are two excitation modes in QCR based techniques: first, the mechanical excitation (QCR is typically attached to a dither piezo and the mechanically induced piezoelectric voltage is measured) and second, the electrical excitation mode (both electromechanical and mechanic-electrical piezoeffects are used for excitation and detection, respectively). The mechanical excitation mode is more popular because of the simplicity of the principle. However, one of the main advantages of the symmetrical quadruple configuration of the tuning forks—the immunity to environmental mechanical and electrical distortions, higher Q factor—in the mechanical excitation mode is sacrificed. The so-called qPlus sensor3 can be seen as limiting case: here one prong of the TF is fixed to a large substrate and the free prong with a tip attached is now a mechanical dipole. Our experience with an electrical QCR used in the self-excitation mode showed no ringing problems using a proper designed circuit. The change of Q factor through tip-sample interaction by operating in environmental conditions is relatively low and may be taken in account. Nevertheless, the first experiments with qPlus sensor were successful, yielding atomic force microscopy (AFM) images of silicon with atomic resolution.4

This example emphasizes the potential of QCR. It seems plausible that in the electrical excitation mode the quadruple symmetry of the tuning fork or the needle quartz positively manifests in the ultimate sensitivity, stability against acoustic and electromagnetic interferences, and stability in time. Consequently, by using a self-excitation high-resolution QCR sensor a SPM system with less expensive acoustical and electromagnetic (em) isolations can be developed. Very stiff QCRs such as the needle quartz or the trident tuning fork driven at subnanometer amplitudes are particularly appealing for use in ambient conditions. Mass balancing of the QCR prongs should be done.

Usually a SPM based on QCR sensors uses a low-noise preamplifier, an industrial lock-in amplifier, and a frequency synthesizer. For example, in this way atomic resolution in ambient conditions with a trident5 and a needle6 QCR is

a)Electronic mail: jersch@uni-muenster.de
b)Electronic mail: maletzk@uni-muenster.de
c)Electronic mail: fuchsh@uni-muenster.de
achieved, Ferrara\(^7\) introduced a simple, cheap, and compact amplitude controlled oscillator based on 32 kHz TF for contact lateral force microscopy.

Here we introduce a high performance electronic interface based on an amplitude controlled bridge oscillator and a phase-locked loop (PLL) frequency demodulator, applicable for noncontact SPM measurements with a QCR platform at frequencies from 10 kHz up to 10 MHz.

**OPERATION PRINCIPLES**

If mechanical properties of QCR are represented by the impedance of an equivalent series circuit (with elements \(L, C,\) and \(R_s\)), the simplified characteristic equation (without considering noise and parasitic capacitance) of the self-excited Barkhausen loop shown in Fig. 1 is

\[
\dot{x}^2LC + xC(R_s - GR_f) + 1 = 0,
\]

with \(G\) representing the variable gain of the automatic gain control (AGC) stage and \(R_f\) the transimpedance resistance of the low-noise amplifier (\(A\)). It can be seen easily that in the excited steady state \((R_s = GR_f)\) the dissipative and conservative interactions are fully separated in a first approximation. Indeed, changes of the \(R_s\) value through the dissipative interactions \(\Delta R\) are immediately compensated through the corresponding gain changes \(\Delta G\). Thus, the second term in the equation is zero and the changes in AGC voltage represent the dissipation changes. Conservative interactions modifies the spring constant of the sensor (the \(C\) value in the equivalent resonance circuit), and resulting in a frequency change \((/f = 2\pi/LC)\) originating from the tip-sample interactions in force measurements. Another mechanism of frequency variations involving changes in effective oscillating mass of the sensor corresponds to the \(L\) value changes. A closer look on the interplay between tip-sample interactions, QCR impedance, measurement, and electronic parameters shows a more complex picture.\(^8\)\(^9\)

If optimal imaging and circuit parameters are used, the noise limit at a given tip-sample interaction is defined by the physical parameters of the QCR (dissipative resistance, material constants, and dimensions). Consequently, the analysis of the noise limit is similar to that in the AFM self-excitation mode.\(^10\)\(^12\)

![Bridge oscillator principle. Oscillation condition is achieved if \(R_2/R_1 = R_f/R_{QCR}\).](Image)

Note that application and analyses of oscillators in quartz crystal microbalance technique are similar and were introduced long before tuning fork sensors in SPM techniques were introduced. The main difference is that in quartz crystal microbalance technique tiny changes in the effective oscillating mass of the sensor are measured rather than forces as in the case of a SPM. Reference 13 gives good introduction and overview of quartz crystal microbalance technique.

**BRIDGE OSCILLATOR CIRCUIT DESCRIPTION**

The design of suitable interface circuits for QCR requires special circuit configurations, which cannot be obtained by simply modifying standard applications. An appropriate interface electronic should deliver fast and precision QCR oscillation amplitude and frequency (or phase) data in order to investigate dissipative and conservative tip-sample forces. Parallel capacity compensation and a grounded QCR (for minimizing parasitic effects and EM interferences and allowing the operation in conductive liquids) are further requirements. Miniaturization and component selection are also crucial for minimizing parasitic effects within the electronic circuit.

We present a precision bridge oscillator circuit with grounded QCR and capacitance compensation. The principle of the bridge oscillator is shown in Fig. 2. Figure 3 shows a variant of the bridge oscillator circuit with Analog Devices (US)\(^14\) elements used. Stable oscillation amplitudes from approximately ten rms of the QCR mechanical noise level up to some nanometers are possible. Low-noise wideband operation amplifiers in transimpedance stage (such as AD8066, AD8067 from Analog Devices or OPA2380, OPA656, and OPA657 from Texas Instruments) allow high performance operation with various QCRs. Note that high frequency elements (AD8067, OPA657, and AD603 bandwidth \(\geq 100\) MHz) in the circuit that operate at relatively low frequency \(\leq 1\) MHz should be used to obtain low phase delay and noise. Output signals of this circuit are the amplitude of the oscillation and the sinusoidal oscillation signal itself.

To provide low current noise the value of the feedback resistor \(R_1\) is approximately one order of magnitude higher than the dissipative resistance of the QCR. The noise of the transimpedance amplifier (input current noise of \(\leq 10\) pA/Hz\(^1/2\) and input voltage noise of \(\leq 10\) nV/Hz\(^1/2\)) is at room temperatures lower than the noise of the feedback resistor and the thermal noise of the dissipative resistance of the QCR. The capacitance compensation is achieved through the variable elements \(C_1\) and \(R_2\). The gain control loop based on a voltage-controlled amplifier (VCA) (AD603) consists of the rectifier (IC1B and IC2A); the proportional-integral (PI) regulator with reference comparator (IC2B) and the adjustable reference voltage source (LM4431, R14) provides stable oscillating amplitude in the bridge circuit. The
amplified control signal on pin 8 IC2C is used as measure for the dissipative resistance of the QCR. The drive amplitude of the QCR can be adjusted with the potentiometer R14. A second order bandpass filter on IC2D improves the output sinusoidal signal. Finally, the compensation capacity C1 should be tuned roughly with C1, fine with potentiometer R2 to achieve the lowest value of the gain control signal.

The most expensive part in the circuit is the gain control stage on VCA AD603 or VCA810. Typically, for gain control in voltage-controlled oscillators cheap field effect transistors FETs are used, e.g., in SPM applications. However, FETs suffer on strong temperature dependence and nonlinearity resulting in low precision of data obtained.

We have miniaturized the layout in order to locate the amplifier at the base of the QCR to reduce interferences and parasitic capacitance due to the cable connecting the sensor to the circuit. The used precision elements reduce the complexity of the circuit. The device is mounted on a 27×35-mm-sized double-sided printed circuit board (PCB) and placed in a shield box. Both lateral and vertical force measurement configurations can be realized by appropriate attachment of the tip to the QCR prong.

**DATA ACQUISITION**

The signal amplitude, the resonant frequency, and the gain control signal are the measurable data in our circuit. With the knowledge of the electromechanical coupling constant and the dimensions of the used QCR, the mechanical tip-sample interaction parameters can be estimated from the electrical measurements in usual way (e.g., see review article). For example, from the amplitude signal and the gain control signal it is easy to calculate the current through the QCR and the dissipative resistance of the QCR. The mechanical oscillation amplitude of the QCR and mechanical dissipation can be estimated straightforwardly.

The frequency shift Δf and the tip-sample force gradient Δk at small oscillation amplitudes are in a particularly simple connection: Δk=2kΔf/f (k is the force constant and f the resonance frequency of the used QCR). For precision frequency measurements, we developed a simple PLL demodulator circuit based on a 74HC4096 phase comparator (an improved variant of the well-known CD4046) and special voltage controlled crystal oscillator (VCXO) (AXTAL GmbH, Mosbach, Germany) with an extremely wide pulling range. The frequency resolution of our demodulator was better as 0.1 Hz at all used frequencies, sufficient for SPM applications. Tests with 32, 100, and 200 kHz TFs and 1 MHZ needle quartz (Fig. 4) showed the high performance of the demodulator circuit. For example, by using the 1 MHz needle quartz with a force constant of k≈400 000 N/m the minimal detectable force gradient Δk is <0.1 N/m, quite enough for nondestructive scanning on relative soft samples.

**FIG. 3.** Schematic of the bridge oscillator circuit. Integrated circuit destinations: IC1A, transimpedance amplifier in bridge connection; AD603, voltage controlled amplifier; IC1B, full rectifier; IC2B, comparator and PI regulator; LM4431, voltage reference; IC2D, bandpass filter; IC2A and IC2C, operational amplifiers.

**FIG. 4.** Various tuning forks (100, 32, and 200 kHz) and 1 MHz needle quartz tested with developed circuit.
The data are comparable with the previous results obtained with the 1 MHz needle quartz in an Omicron ultrahigh vacuum system. It should be noted that in measurements on soft samples at ambient environment, images obtained in the dissipative channel (AGC control signal) are generally of higher quality as those obtained with the frequency modulation mode.

The developed electronic is applicable with nearly all industrial SPMs with optional signal inputs. For example, we used two external analog inputs display dissipative and conservative data channels in an NT-MDT system (Moscow, Russia) and Solver SPM as well as in a homebuilt scanning near-field optical microscopy (SNOM) based on PI (Waldbronn, Germany) XYZ piezosystem. In the latter system, the complex self-excitation circuit with dither piezo was successful replaced by the described circuit.

FORCE MICROSCOPY IMAGES

Tapping mode force image made on a graphite surface [highly oriented pyrolytic graphite (HOPG)] in ambient conditions by using a 1 MHz needle quartz with a standard cantilever tip attached is shown in Fig. 5. On a $4 \times 4 \mu m^2$ area monoatomic terraces are resolved in the dissipative mode. The noise in cross section line (Fig. 6) is dominated by the mechanical distortions of our SPM (NT-MDT) resolution. Furthermore, in Fig. 7 we present images obtained by using of developed circuits in the SNOM distance control. As a force sensor, the relatively large tetrahedral glass probe attached on a 32 kHz tuning fork is used. In order to test the capability in measurements on soft samples we used monomolecular dipalmitoylphosphatidylcholine (DPPC) Langmuir-Blodgett (LB) strips. Figure 8 shows that the interaction is gentle enough to make nondestructive images on this fragile samples. Multiple scans across LB strips revealed no changes.

SUMMARY

We have reported on a self-excitation interface circuit for force detection based on different QCRs. A bridge oscil-
lator configuration with grounded QCR for precision distance control was developed and tested in various applications (SNOM, SCM, and other). An optional precision PLL demodulator is developed for the detection of frequency shift. Frequency shift and QCR loss representing conservative and dissipative interactions in noncontact (NC)-AFM measurements are used to demonstrate the device capability. Highly resolved graphite and LB stripe images in AFM application using the needle quartz and SNOM applications using 32 kHz TF demonstrate the performance of the device. It is shown that a very stiff mechanical sensor allows probing very soft samples without damage. The inherent fast operation in both dissipative and conservative force measurement modes and the capability of applying the sensor in liquid environment open wide applications.

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14 A slightly lower temperature stability has another variant of the circuit with the gain control stage on VCA810 (from Texas Instruments, TX).