A simple setup to simultaneously measure the resonant frequency and the absolute dissipation factor of a quartz crystal microbalance

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An experimental setup is described that can simultaneously measure the absolute dissipation factor and the resonant frequency of a short-circuited quartz crystal microbalance. The crystal is driven at approximately its resonant frequency by a signal generator which is intermittently disconnected by a relay, causing the crystal oscillation amplitude to decay exponentially. The decay is measured using a ferrite toroid transformer. One of the crystal leads is fed through the center of the ferrite toroid and thereby acts as the primary winding of the transformer. The secondary winding of the transformer is connected to a digitizing oscilloscope which records the decay of the crystal oscillation. From the recorded decay curve, the absolute dissipation factor (calculated from the decay time constant) and the series resonant frequency of the freely oscillating crystal are obtained. Alternatively, the dissipation factor and resonant frequency can be measured for the crystal oscillating under open-circuit conditions, i.e., in the parallel mode. The measurements are automated. © *1996 American Institute of Physics.* [S0034-6748(96)00209-2]

I. INTRODUCTION

The quartz crystal microbalance (QCM) is commonly employed as a sensor in, e.g., film deposition and film growth in vacuum,[1] and in liquids.[2] It is also used in measurements of liquid density and viscosity,[3] etc. In its most common implementation, the QCM is used as the frequency-determining element of an electronic oscillator circuit, and the changes in the oscillation frequency are recorded. However, there are several disadvantages of using an electronic oscillator that only allows the measurement of resonant frequency: (i) the frequency shift of an oscillator circuit always contains some admixture from the energy supplying elements which can interfere with the ''true'' frequency shift due to, for example, a mass increase;[4] (ii) the measured change in QCM resonant frequency depends on the phase shift introduced by the electronic oscillator, which is in most cases not known;[5] (iii) there are many situations where a measurement of (the changes in) the absolute dissipation factor (i.e., the damping) of the crystal oscillation would be valuable as a complement measurement.[2,4,6]

These problems could be addressed by using an impedance analyzer to measure the impedance magnitude and phase of the QCM, instead of using an electronic oscillator.[7] However, an impedance analyzer is rather expensive. Our research group has recently published a simple setup which overcomes many of the disadvantages of the electronic oscillator.[8] It allows for the simultaneous measurement of the parallel resonant frequency, f_p, and the corresponding dissipation factor, D_p, with a repetition rate of $\sim$1 Hz. No phase shift is introduced by the electronic driver since the crystal is freely oscillating (disconnected from the electronic driver) during the measurement. However, there are situations where it would be desirable to also, or as an alternative, measure the series resonant frequency, f_s, and the corresponding dissipation factor, D_s, of the QCM. For example, there have been reports that when one side of the crystal is facing a liquid f_p, but not f_s, is affected by the conductivity of the liquid.[9,10]

It should be noted that the majority of the equations used to relate the frequency and dissipation factor responses of a QCM to a mass or liquid loading have been derived assuming (often implicitly) that the crystal is oscillating under short-circuit (series mode) conditions. In this work, we describe a simple setup that allows the simultaneous measurement of the series resonant frequency and the series dissipation factor. The setup also allows the measurement of the parallel resonant frequency and dissipation factor. In the latter case, the setup loads the crystal with a capacitance of approximately 19 pF.

The principle of the measurement is that when the driving power to a piezoelectric resonator is switched off at time $t=0$, the voltage over the resonator, $U(t)$, decays as an exponentially damped sinusoidal:

$$U(t)=A_0 e^{-t/\tau}\sin(2\pi f t+\phi), \quad t\geqslant 0, \tag{1}$$

where A_0 is the amplitude at $t=0$, τ is the decay time constant, and ϕ is the phase. The dissipation factor is related to the decay time constant as

$$D=\frac{1}{\pi f \tau}. \tag{2}$$

II. EXPERIMENTAL SETUP

In brief, f and D measurements are made by periodically disconnecting the oscillating crystal from a driving circuit (an HP 33120A arbitrary waveform generator) by a computer controlled relay (Clare, MSS-2). The decay of the QCM oscillation is recorded on a digitizing oscilloscope (Tektronix, TDS 410A). The decay curve is subsequently transferred, via GPIB, to a computer and a numerical fit of Eq. (1) is performed. From the fit, both the resonant frequency and the

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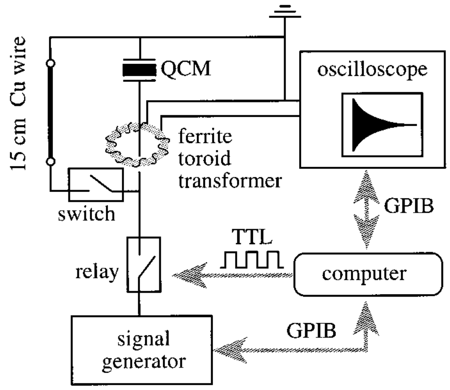

FIG. 1. A schematic illustration of the experimental setup. The crystal is driven by a signal generator approximately at its series resonant frequency. The crystal decays in the parallel or series mode when the switch is open or closed, respectively.

dissipation factor are obtained simultaneously. An f and D measurement (including curve fitting) takes approximately 1–2 s.

In the previous study, a high impedance unity gain buffer (Burr-Brown 3553), connected in parallel with the QCM, was used to probe the decay of the crystal oscillating in the parallel mode.[8]

In the present study we describe a simple setup that can be used to measure *both* the series and the parallel resonant frequencies, f_s and f_p, respectively, and the corresponding absolute dissipation factors. The setup is schematically shown in Fig. 1.

A small ferrite toroid (Philips 3F3) is used as a current transformer. One end of the secondary leads was connected to a 50 Ω input of a digitizing oscilloscope and the other one was grounded (together with one of the QCM electrodes) at the oscilloscope. The configuration of the transformer depends on the mechanical load of the crystal (vide infra). When the crystals were oscillating in air, we used one turn on the primary side (achieved by simply threading the lead between the relay and the crystal through the hole in the ferrite toroid) and ten turns on the secondary winding. With this configuration, the load of the measurement setup on the crystal, R_{load}, is approximately 0.5 Ω. In liquids, we used 4 turns on the primary side and 10 on the secondary side of the transformer which increased R_{load} to approximately 8 Ω. As a comparison, the motional resistance is typically 5–30 Ω for a QCM oscillating in air, and 200–400 Ω when the crystal oscillates with one side in contact with water.

The signal generator is approximately tuned to the series resonant frequency. When the relay is opened the crystal oscillation frequency shifts to the crystal's series or parallel resonant frequency depending on whether the switch is closed or open, respectively. The measured f and D (series or parallel mode) do not depend on the exact driving frequency as long as it is close enough to f_s to produce a good output signal. The output signal is largest when the driving frequency equals f_s. (By sweeping the frequency of the signal generator while measuring the amplitude of the output signal and the phase between the output and signal generator signals, it is possible to obtain an impedance plot very much like that of an impedance analyzer.) A signal generator was used instead of an ordinary electronic oscillator so that the crystal can be driven even when short-circuited with a copper wire, as described below.

The switch consists of a BNC to double a 4 mm socket adapter (Greenpar) with the sockets short-circuited by a 15 cm long, 2 mm thick copper wire. The copper wire has a self-inductance, L_s, of approximately 90 nH. The switch is closed by connecting this adapter to a BNC plug between the relay and the toroid transformer. (The BNC adapter can, of course, be replaced by a relay in order to operate it by the computer.) The BNC contacts used to connect the switch, the relay, and the crystal together adds a load capacitance, C_s, of $\sim$19 pF.

In the previous setup, a dc offset voltage appears over the crystal whose magnitude depends on the voltage over the crystal when the relay is opened.[8] This dc offset either has to be filtered out or it has to be included in Eq. (1) as an additional fitting parameter. In the present setup this is not a problem since the transformer does not transmit dc voltages. (The opening of the relay produces a transient of approximately 10 ns duration which is transmitted to the oscilloscope. This transient is avoided by starting the data acquisition 0.1 ms after the relay opens.)

The load on the crystal during measurement (i.e., after the relay has been opened) depends on whether the switch is open or closed (i.e., whether the BNC adapter is disconnected or connected, respectively). When the switch is closed (series mode), the capacitance C_s can be ignored since $\omega L_s \ll (\omega C_s)^{-1}$, and the load on the crystal is therefore R_{load} in series with L_s. When the switch is open (parallel mode), the load consists of R_{load} in series with C_s.

The influence of the setup on the resonant frequencies and the dissipation factors can be estimated as follows.[2] In the series mode, the resonant frequency is, to a good approximation, given by

$$f_s = \frac{1}{2\pi\sqrt{C_1(L_1+L_s)}}, \tag{3}$$

where C_1 and L_1 are the crystal's equivalent circuit motional capacitance and inductance, respectively, and are illustrated in Fig. 2. Also shown in Fig. 2 are the crystal's motional resistance, R_1, and parallel capacitance, C_0. In the parallel mode of the setup, the resonant frequency is given by

$$f_p = \frac{1}{2\pi\sqrt{L_1\dfrac{(C_0+C_s)C_1}{C_0+C_s+C_1}}}. \tag{4}$$

The dissipation factor is the same for both series and parallel mode (provided that the crystal is operating in a nonconducting environment) and is given by

$$D = \frac{R_1+R_{\text{load}}}{2\pi f_s L_1} \approx \frac{R_1+R_{\text{load}}}{2\pi f_p L_1}. \tag{5}$$

From Eqs. (3) and (4), it is possible to estimate how much the setup influences the measurement in the series mode. The unperturbed f_s, f_p, and D values are given from Eqs. (3), (4), and (5), respectively, with the load elements L_s, C_s, and

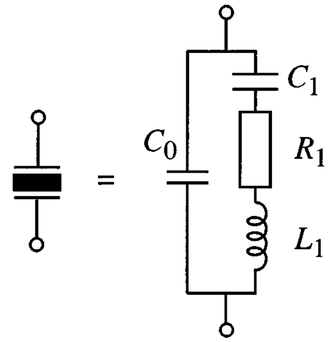

FIG. 2. The equivalent circuit of a QCM near resonance. The equivalent circuit elements L_1, C_1, and R_1 represent the mechanical properties of the crystal and constitute the so-called motional arm. The parallel capacitance C_0 is due to the capacitance caused by the crystal electrodes and constitutes the so-called parallel arm of the equivalent circuit.

TABLE I. The resonant frequencies and dissipation factors for four crystals measured in the series and parallel modes. The series resonant frequency is lower than the parallel resonant frequency for all four crystals as expected. The difference $f_s - f_p$ increases with increasing resonant frequency, also as expected.

Crystal	f_s (Hz) ±1 Hz	$f_p - f_s$ (Hz) ±2 Hz	D (10^{-6}) $\pm 10^{-7}$
1	1 843 006	306	16.0
2	5 240 151	2024	8.5
3	9 988 286	5659	8.6
4	14 740 805	7251	11.7

R_{load} set to zero. As an example, we consider a typical AT-cut, 10 MHz quartz crystal with L_1=9 mH, C_1=28 fF, C_0=5 pF, and R_1=5 Ω. Using Eqs. (3) to (5), we find that the measured resonant frequencies of the series and parallel mode are 50 Hz and 22 kHz lower than the unperturbed resonant frequencies, respectively. The dissipation factor is increased by 9×10^{-7} (10%), due to the influence of the experimental setup.

III. PERFORMANCE

We have successfully tested the setup on crystals with resonant frequencies ranging from 1.8 MHz to 14.7 MHz. Four examples of crystals with different resonant frequencies are listed in Table I.

In most QCM sensor applications, the stability of the measurement is usually much more important than an offset introduced by the measurement setup. The stability of the setup in air is illustrated in Fig. 3 which shows changes in f_s, f_p, D_s and D_p as a function of time for a 10 MHz AT-cut crystal (crystal 3 in Table I). The stability in resonant frequency was better than 0.1 ppm and the stability in dissipation factor was better than 10^{-7} over a period of 15 minutes for the crystals tested. We did not observe any long term drifts in resonant frequency and dissipation factor as long as the temperature of the QCM was held constant.

The setup has also been tested with one side of the QCM exposed to a liquid. The motional resistance, R_1 is greatly increased when the crystal is submerged into a liquid (typically by a factor of ~100). This means that the current that flows through the crystal, and thereby through the primary winding, decreases. This causes a reduction in signal and, consequently, in signal-to-noise ratio. To increase the signal a ferrite toroid transformer with 4 turns on the primary winding and 10 turns on the secondary winding was used. This increases the absolute load on the crystal, but since the relative increase in R_1 is even larger, the relative load will actually decrease. The short term stability of a 5 MHz, AT-cut crystal with one side facing water was approximately ±2 Hz in *f*, and the stability in *D* was approximately $\pm2\times10^{-6}$. No long term drifts in *f* and *D* were observed if the temperature of the liquid cell was held constant. (The noise can be reduced by mixing the output signal from the decaying crystal with a stable reference signal and measuring on the difference frequency. Using mixing with a difference between the reference signal and the decaying crystal of approximately 0.1 MHz, we found that the short term stability in *f* and *D* was improved 2 and 4 times, respectively, for a 5 MHz crystal operating with one side in water.)

Instead of measuring the current through the crystal, as done in the setup shown in Fig. 1, it is possible to measure the voltage directly over the crystal in order to estimate f_s and D_s, as illustrated in Fig. 4. This latter setup has the advantage of effectively render R_{load}=0 but we did not achieve as low noise with this setup as with the one shown in Fig. 1.

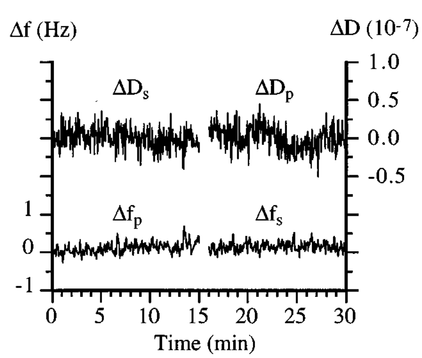

FIG. 3. Variations in f_s, f_p, D_s, and D_p as a function of time for a 10 MHz AT-cut crystal (crystal 3 in Table I). The switch was open during the first 15 minutes (parallel mode) and closed the remaining time (series mode). *D* did not change significantly when the mode of operation was changed from the series to the parallel mode, provided that the crystal was operated in air and the crystal drive level was kept constant.

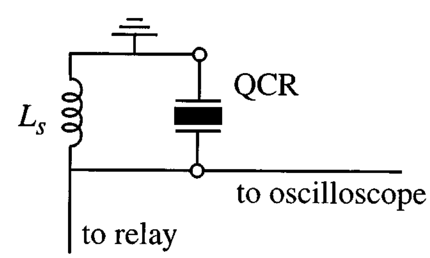

FIG. 4. A detail from the setup from Fig. 1 modified so that the voltage over the crystal can be measured.

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