



## ADVANCED ELECTRONIC INTERFACES FOR THE MONITORING OF AT-CUT QUARTZ CRYSTAL RESONATORS USED AS ACOUSTIC SENSORS IN FLUID MEDIA

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### ABSTRACT

The classical quartz crystal microbalance (QCM) is no longer only a microbalance; it has got a place as an acoustic sensor in a broad range of applications such as: fluid physical characterization, viscoelastic study of polymers, charge transfer analysis in electrochemical processes, and detection of biological components in fluid media, among other applications. In this paper, the basic operation of an AT cut quartz crystal resonator is extended to the fluid environment. In these applications the resonator is submitted to a heavy load which strongly affects the sensor response, making especially difficult the characterization of the main sensor parameters. The problem associated with the electronic interfaces for sensor characterization is introduced along with a brief reviewing and some recent improvements. After this description, an improved electronic interface is introduced in detail. The design is an interface based on a phase locked loop system which permits an accurate monitoring of the series resonant frequency and the motional resistance of the quartz crystal resonator sensor. A continuous and automatic compensation of the sensor parallel capacitance makes this possible. The report of experimental results shows the benefit of the new system, especially for heavy load QCM applications.

### INTRODUCTION

The thickness shear mode (TSM) quartz crystal resonator (QCR) has been extensively used as quartz crystal microbalance (QCM) sensors in gaseous media [1]. However the limited penetration depth of the shear acoustic wave in liquid [2] has extended its applications to fluid environment for detecting different processes which occur in the interface between the sensor and/or the coating and the liquid medium. The complete physical description of a viscoelastic load in contact with the QCR [3] has allowed the study of mechanical properties of different materials coated on the surface of the sensor, like viscoelastic properties of polymers [4,5]. In these cases concepts like acoustically thin or acoustically thick coatings are of fundamental importance [6]; in case of a thick viscoelastic film in contact with a liquid, a complete characterization of the sensor together with alternative techniques are necessary for a comprehensive explanation of certain phenomena involved during the experiments [7], on the contrary, for acoustically thin films, great simplifications can be done in the physical model [7,8] and the extraction of the physical properties of interest can be done by a simple characterization of appropriate parameters of the sensor. These simplifications can be done in a great deal of applications such as: fluid physical characterization, for both Newtonian and/or viscoelastic fluids [7], charge transfer analysis for studying the behaviour, for instance, of conductive polymers in electrochemical processes [9], detection of immunoreactions and the development of biosensors [10], etc. However, even in the simplest cases, sensor characterization is performed through electronic interfaces that must be able to measure and to monitor, even continuously, appropriate sensor parameters which are related to the physical properties to be evaluated.

In this article a brief description of the characteristic properties of the TSM-QCR sensor, specially for simple cases, is introduced as a starting point for determining appropriate

parameters of interest in the sensor characterization in a great deal of applications. Once the parameters of interest are established a summary of the electronic interfaces habitually used for sensor characterization is introduced with the main advantages and drawbacks. Special attention will be paid to the use of oscillators as drivers for QCR sensors and to the associated problems. Finally some recent oscillator-like operating circuits which try to avoid the disadvantages of actual oscillators are introduced. These circuits are based on parallel automatic capacitance compensation (ACC) techniques.

## THEORY

The expression of the admittance  $Y$  of TSM QCR sensors, is given by [11,12]:

$$Y = j\omega C_0^* + \frac{1}{Z_m} \quad (\text{Eq. 1})$$

where  $C_0^*$ , is the so-called “static” capacitance and  $Z_m$  is the impedance of the so-called motional branch (Fig.1a). The static capacitance arises from electrodes located on opposite sides of the dielectric quartz resonator  $C_0$  and from parasitic capacitances external to the sensor  $C_p$ :  $C_0^* = C_p + C_0$ . The motional branch arises from electrically excited mechanical motion in the piezoelectric crystal and, around resonance, can be modelled by the series lumped-element equivalent circuit,  $L_1, C_1, R_1, Z_L^E$  shown in Fig.1b, where  $L_1, C_1, R_1$  refer to the unperturbed sensor and  $Z_L^E$  is a complex impedance representing the contribution of the load.

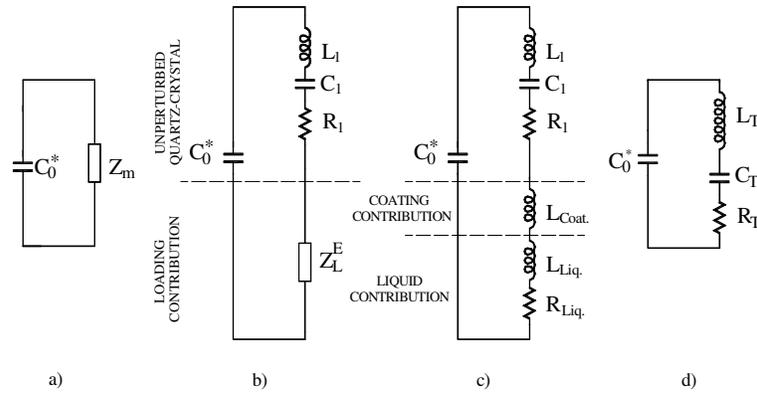


Figure 1.-Lumped-element equivalent circuits of the sensor

The base that makes the QCR useful as sensor is the relationship between the electrical complex impedance  $Z_L^E$  and the physical parameters of the load. Thus, by appropriate characterization of the electrical parameters of the sensor, it is possible to extract the physical parameters of the coating. For most of practical cases the electrical impedance  $Z_L^E$  is proportional to the surface acoustic load impedance  $Z_L^A$  through Eq.2 [13].

$$Z_L^E = \frac{N\pi}{4k_0^2 \omega_s C_0 Z_q} Z_L^A \quad (\text{Eq. 2})$$

where  $N$  is the harmonic resonance of the QCR,  $k_0$  is the quartz electromechanical coupling factor,  $\omega_s$  is the series resonant angular frequency and  $Z_q$  is the quartz characteristic impedance.

For acoustically thin or rigid coating, the surface acoustic load impedance has only imaginary component, which is proportional to the surface mass density of the coating according to Eq.3 [7,14], and then the electrical impedance has only reactive contribution that can be modelled with an inductance  $L_{Coat}$  as shown in Fig.1c.

$$Z_L^A = j\omega\rho_C h_C \quad (\text{Eq. 3})$$

where  $\rho_C$  and  $h_C$  are the density and thickness of the coating respectively.

For a viscoelastic fluid medium in contact with the sensor, the surface acoustic load impedance has both real and imaginary components described by Eq.4. Thus the electrical contribution of a fluid in contact with the sensor can be modelled by a series lumped-element formed by a resistance  $R_{Liq}$ , and an inductance  $L_{Liq}$  (Fig.1c).

$$Z_L^A = \sqrt{\frac{\rho_L G_L''}{2}} \sqrt{\sqrt{Q_L^2 + 1} + Q_L} + j \sqrt{\frac{\rho_L G_L''}{2}} \sqrt{\sqrt{Q_L^2 + 1} - Q_L} \quad (\text{Eq. 4})$$

where  $\rho_L$  is the density of the liquid,  $G_L'' = \omega\eta_L$  is the viscoelastic shear loss modulus, being  $\eta_L$  the viscosity of the fluid,  $Q_L = G'/G''$  is the reverse of the loss tangent of the fluid, being  $G'$  the viscoelastic shear storage modulus. As it can be noticed, when the fluid is Newtonian,  $Q_L = 0$  and the real and imaginary parts of the impedance have the same magnitude which is depending on the density-viscosity product.

In general, for the purposes of this paper, it is enough to consider the sensor modelled by the lumped-element equivalent model in Fig.1d.

### PROBLEM ASSOCIATED WITH ELECTRONIC INTERFACES FOR SENSOR CHARACTERIZATION

Impedance or Network Analyzers are typically used in laboratory for a complete electrical characterization of the sensor around resonance and mainly for static or slow response experiments. These systems characterize the sensor in isolation, i.e., its response is not altered by the external circuitry and they permit the exclusion of parasitic influences by calibration. However, the high costs and large dimensions of the equipment, and even more important the difficulty of performing a fast monitoring of the sensor parameters, prevent their use for sensor applications. On the other hand, oscillators are suitable for sensor applications, principally due to its fast operation, to the low expense of circuiting and to the mayor adaptability for remote or *in situ* measurements, but great drawbacks remain with the oscillators currently used for QCM applications in liquid media.

Oscillators' output frequency depends on the specific loop gain and phase oscillating conditions; therefore different oscillators can provide different output frequencies for the same experiment depending on the specific designed oscillating conditions. The problem is qualitatively shown in Fig.2 which depicts the typical admittance circle of a quartz sensor subject to different loads.

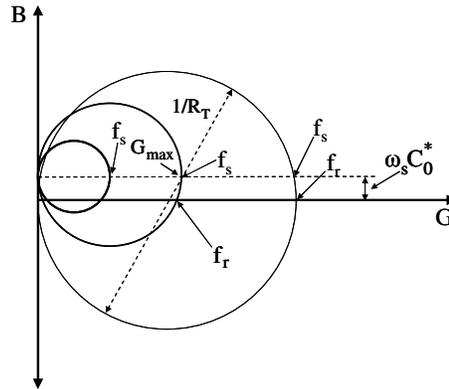


Figure 2.-Admittance circle of a QCR sensor under acoustic and dielectric loading

The upward shift of the circle is proportional to  $C_0^*$ , and the diameter inversely related to  $R_T$ . The zero-phase resonant frequency  $f_r$  is significantly different from the series resonant frequency  $f_s$ , which corresponds to the maximum of  $G$ , i.e. the real part of the admittance.

The admittance phase at  $f_s$  is:

$$\Phi(f_s) = \arctan(2\pi f_s R_T C_0^*) \quad (\text{Eq. 5})$$

It can be observed that the phase is not univocally defined because it is a function of the loading conditions through  $R_T$  and  $C_0^*$ . On the other hand, the cancellation of the parallel capacitance [15,16] reduces the electrical model of the sensor in Fig.1d to the series motional branch only, and then the series motional resonant frequency is univocally determined at the zero-phase condition of the motional branch.

### CIRCUIT DESCRIPTION

The concept behind the design is the same introduced elsewhere [16]. The new approach, shown in Fig.3, was partially introduced elsewhere [15] for manual capacitance compensation. In the present case two phase locked loops (PLL) are used for both frequency tracking and parallel capacitance compensation. The PLL in charge of the sensor series resonant frequency tracking is based on a phase-frequency detector (PFD) as it is also the case for capacitance compensation. Therefore, an easier and, in principle, a more accurate calibration of the PLLs can be performed in this new configuration by following the procedure introduced elsewhere [15].

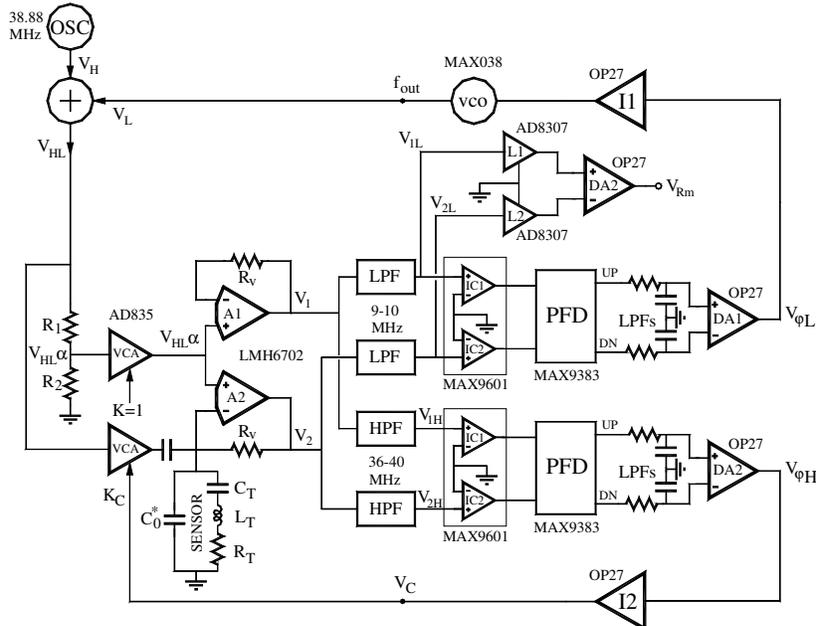


Figure 3.-Circuit block diagram for the automatic capacitance compensation system

The magnitudes governing the operation of the system are the phase-shifts between the signals  $V_1$  and  $V_2$  at the two frequencies,  $f_H$  and  $f_L$ . The phase-shift at the lower frequencies around the series resonant frequency of the sensor controls the PLL in charge of the frequency tracking, while the phase-shift at the auxiliary higher frequency, where only capacitive behaviour of the sensor is expected [15], controls the PLL in charge of the parallel capacitance compensation. Therefore, the equation governing the control of the system is:

$$V_{2HL} = (1 + R_v Y_T + j\omega R_v (C_0^* - C_c)) V_{1HL} \quad (\text{Eq. 6})$$

where  $V_{1HL} = V_{HL} \alpha$ ;  $C_c = [K_C/\alpha - 1]$ ;  $\alpha = R_1/(R_1 + R_2)$ ; and  $Y_T = [j\omega L_T + R_T + 1/j\omega C_T]^{-1}$ .

The subindex  $HL$  in the previous equations means that the voltage waveform considered is the sum of the two sinusoidal signals  $V_H$ , with fix frequency  $f_H$ , and  $V_L$  with frequency  $f_L$  generated by the VCO around the series resonant frequency of the sensor.

At the auxiliary frequency,  $f_H$ , where only capacitive behaviour of the sensor is expected, Eq. 6 is reduced to:

$$V_{2H} = (1 + j\omega R_v (C_0^* - C_c)) V_{1H} \quad (\text{Eq. 7})$$

and the phase shift between the signals  $V_{2H}$  and  $V_{1H}$  is given by:

$$\Phi(V_{2H}, V_{1H}) = \arctan(2\pi f_H R_v C_r) V_{1H} \quad (\text{Eq. 8})$$

where  $C_r = C_0^* - C_c$  is the residual uncompensated parallel capacitance.

As it can be noticed when  $C_r = 0$  the phase shift is zero and the differential amplifier DA2 gives zero voltage at its output that makes the integrator I2 to maintain a continuously stable dc voltage at its output. This is the only stable condition for the loop out of saturation. The output  $V_C$  of the integrator I2 can be used to monitor the changes in the parallel capacitance of the sensor and also for a continuous monitoring of its magnitude.

At the frequency  $f_L$ , and assuming that the parallel capacitance has been compensated ( $C_r = 0$ ), Eq. 6 is reduced to:

$$V_{2L} = [1 + R_v G_T + jR_v B_T] V_{1L} \quad (\text{Eq. 9})$$

where  $G_T = R_T / (R_T^2 + X_T^2)$ ;  $B_T = -X_T / (R_T^2 + X_T^2)$  and  $X_T = \omega L_T - 1/\omega C_T$

As it can be noticed from Eq. 8 signals  $V_{2L}$  and  $V_{1L}$  will be in phase when  $X_T$  is null and this only happens at series resonant frequency,  $f_s = (2\pi L_T C_T)^{-1/2}$ , which will be the locking condition of the tracking frequency loop.

## EXPERIMENTAL RESULTS

The circuit was implemented in a four-layer board by using the integrated circuits indicated in Fig. 3 and the following relevant component values:  $R_1=R_2=50\Omega$ ,  $R_v=237\Omega$ ,  $C = 18\text{pF}$ . After the initial calibrations procedures indicated elsewhere [15] several experiments with 9 and 10 MHz sensors, one-face in contact with bi-distilled water, were performed. For each sensor external parallel capacitances of different magnitudes were added in order to check the effectiveness of the automatic parallel capacitance compensation of the system. The series resonant frequency of the sensor, one-face in contact with water, was previously measured, for each value of the added capacitor in parallel with the sensor, by measuring the corresponding maximum conductance frequency with an impedance analyzer; afterwards the locking frequency of the system was monitored in two different cases: without connecting the ACC loop and with the ACC loop connected. The results are shown in Fig. 5 for 9MHz quartz crystal.

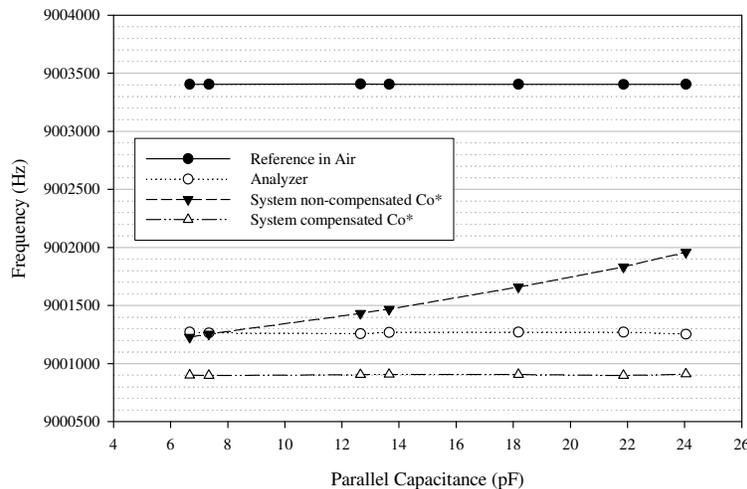


Figure 4.- Experimental results showing the effectiveness of the ACC.

As it can be noticed, a change in the parallel capacitance can produce a change in the oscillating frequency of an oscillator working at zero-phase condition, as it is the case of the proposed system when the ACC loop is disconnected. However, there are no changes in the series resonant frequency when a parallel capacitance is added to the sensor, as it can be noticed by the maximum conductance frequencies measured with the impedance analyzer. When the ACC loop is connected the parallel capacitance is compensated and the looking frequency is maintained constant in all the cases, as it can be observed in Fig. 4.

## CONCLUSIONS

Automatic Capacitance Compensation Systems are a good alternative to oscillators for fast sensor characterization systems while providing a real-time monitoring of three parameters of interest: the series resonant frequency, the motional resistance and the change in the parallel capacitance of the sensor. This capability makes the ACC systems to be appropriate devices for experiments where changes in the properties of the medium imply a change in the parallel capacitance and/or in the quality factor of the sensor.

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