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A DDS-based multi-harmonic frequency meter for QCM sensor applications

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Abstract

In this paper we present a prototype of a frequency meter based on an ARM processor and a DDS module. The instrument was developed to be used with QCM-sensor based oscillator circuits for in-liquid applications. For each resonance frequency of the QCM sensor, up to 50 MHz, the instrument can generate a reference signal exploiting the DDS module, which is used to perform a heterodyne demodulation of the oscillator signal. The ARM processor is used to on-line change the DDS settings (to track up to three resonance frequencies components with interleaved sampling of the QCM signals with a sampling time of about 1s) and to perform the frequency measurements. The contemporary use of the information from different QCM resonance frequencies will be exploited to enhance the instrument sensitivity while preserving a resolution of about 1 Hz for short term measurements with 10 MHz QCMs. At present, preliminary tests were performed.

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Keywords: Multiple-harmonic oscillator; Quartz crystal microbalance; QCM; Overtone; In liquid measurements.

1. Introduction

The measurement of the frequency shifts of multiple harmonics from quartz crystal microbalance has already shown its potentiality and some systems exploiting this idea were presented in the literature. Using the quartz resonator at multiple-harmonic modes, enhances sensing capabilities because a larger set of parameters can be measured on a single sensor. For multiple-harmonic operations with QCM some front-end circuit were proposed as for instance the switched-mode oscillators.

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In this paper a prototype of a frequency meter based on an ARM processor and a DDS module is presented. A low cost instrument controlled by a PC was developed to be used with QCM-sensor based oscillator circuits for in-liquid applications. For each resonance frequency of the QCM sensor, up to 50 MHz, the instrument can generate a reference signal exploiting the DDS module, which is used to perform a heterodyne demodulation of the oscillator signal. The ARM processor is used to on-line change the DDS settings (to track up to three resonance frequency components with interleaved sampling of the QCM signals, with a sampling time of about 1s) and to perform the frequency measurements. The contemporary use of the information from different QCM resonance frequencies will be exploited to enhance the instrument sensitivity while preserving a resolution of about 1 Hz for short term measurements with 10 MHz QCMs.

Nomenclature

C_0	static capacitance of the quartz, $C_0=\varepsilon_q A/h_q$
A	active area of the quartz
h_q	thickness of the quartz
e_q	piezoelectric constant (e_{26} for an At-cut)
μ_q	shear modulus of the quartz ($\mu_q=c_{66}^D$)
ρ_q	density of the quartz
η_q	quartz viscosity

2. Theory of operations

A QCM operating in liquid is a heavily loaded quartz resonator. The sensor output can be either one of its natural resonance frequencies which are affected by any change in the mechanical system, sensitive both to mechanical loading due to the surrounding medium and to possible adsorbed mass, or the damping factor corresponding to these resonances. Under the assumptions of a thin plate with parallel and indefinite surfaces and of uniform load, a lumped parameter circuit approximating the behavior of the QCM can be used (figure 1) which describes the quartz around the resonance frequencies [1] being only one of the motional arms active around a given resonance frequency, while the others behave as open circuits. In this case the electrical admittance of the QCM can be expressed as:

$$Y_q = j\omega C_0 + \sum_i Y_{mi}, \quad \text{with} \frac{1}{Y_{mi}} = \frac{1}{j\omega C_{mi}} + j\omega L_m + R_{mi} + Z_{l_i} + \frac{1}{j\omega(-C_0)} = R_{Ti} + jX_{Ti}$$

$$C_{mi} = \frac{8Ae_q^{\,2}}{h_q\mu_q i^2\pi^2}; L_m = \frac{1}{8}\frac{\rho_q h_q^3}{Ae_q^{\,2}}; R_{mi} = \frac{\eta_q h_q i^2\pi^2}{8e_q^{\,2}A}; X_{Ti} = \frac{-1}{\omega C_{si}} + \omega L_m + \mathrm{Im}(Z_{li}); R_{Ti} = R_{mi} + \mathrm{Re}(Z_{li})$$

Z_{li} is the equivalent electric impedance representing the acoustic impedance of the load around the i-the resonance frequency.

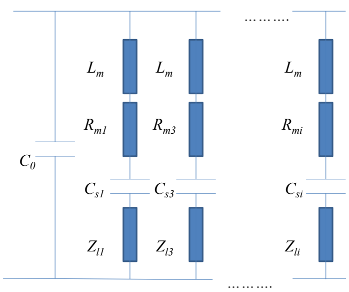

Figure 1 – Equivalent circuit for a QCM

The acoustic, i.e. mechanical, load can be simply inductive in the case of pure and rigid mass adsorption, in the so call e gravimetric regime, or complex when the mass loading is accompanied by damping, such as in liquids. For the model shown in Fig. 1, the i-th series resonant angular frequency, $\omega_{s,i}$ where i is an odd integer, depends only on the electromechanical system and accounts for the mass and stiffness contributions of the load, whereas it is not influenced by the damping effect and by the parallel branch that includes C_0. Note that frequency spacing of harmonics is slightly greater than twice the fundamental due to the presence of the negative capacitance $-C_0$ in the motional arm whose value is constant, irrespective of the harmonic considered.

In case of a quartz in gravimetric regime, i.e a film of thickness h_f and density ρ_f deposited over the quartz, we have $Z_l = j\omega L_l = j\omega\,(h_q^2 \rho_f h_f)/\,(4e_q^2 A)$, and therefore so the series resonance angular frequency of the loaded quartz $\omega^l_{s,i}$ shifts with respect to the one of the unloaded quartz, $\omega_{s,i}$, and we find:

$$\omega^l{}_{s,i} = \sqrt{\frac{1}{C_{si}\left(L_m + L_l\right)}} \approx \omega_{s,i}\left(1 - \frac{L_l}{2L_m}\right) \Rightarrow \Delta f_{s,i} = f^l{}_{s,i} - f_{s,i} = -\frac{2 f_{s,i}{}^2}{i}\frac{\rho_f h_f}{\sqrt{\rho_q h_q}}$$

Typical low cost systems used with QCM for in-liquid applications exploit a simple oscillator in the front-end circuit and an accurate frequency measurement. Oscillators operate at a frequency corresponding to a given Y_q phase, many of them at $\Phi\,(Y_q) = 0°$, so the oscillator output frequency (or frequencies) does not exactly correspond to the series resonance frequency of the motional branch, but depends also on the static electric capacitance, (including the parasitic ones) and on the real part of the quartz electric impedance. Frequency shifts related to different resonant modes can vary in a different way depending on the environment and on the front-end. Hence the measurement of more than one frequency gives more information about the system under study. The idea at the base of this work is to maintain the measurement system simple, and to design a multiple harmonic measurement system providing an extended number of measured parameters, in order to enhance the performance of QCM based system.

3. Developed system

The proposed hardware is a stand-alone, low cost instrument based on an ARM microprocessor (STM32F4) and on a superetherodyne structure obtained by mixing the QCM oscillator signal with a reference signal generated from a high-resolution, programmable DDS (AD9854) capable of sub-Hertz frequency resolution (figure 2).

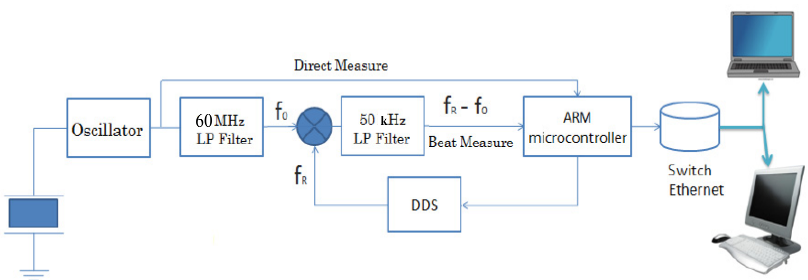

Fig. 1. Block diagram of measurement system.

Fig. 2. Measurement chamber and front-end electronics.

In the proposed measurement instrument the DDS settings can be changed on line in a short time to generate the desired reference frequency without any hardware modification. The measurement system is interfaced via Ethernet and can be set-up and exchange data with a remote host via a TCP/IP protocol. For instance, a Labview VI can manage the measurement campaign from a host PC. The QCM is a 10 MHz AT-cut quartz resonator, which is functionalized to obtain target-selective adsorption. The quartz is placed in chamber suitable for in-liquid applications, and a Meacham oscillator is used as front-end electronics (figure 2). The first 3 harmonics are monitored with interleaved measurements with a sampling time of 1s, so that the three frequency variations can be simultaneously tracked during the target species adsorption.

The theoretical resolution of a quartz in water is well below 1 Hz for all the considered harmonics, but the influence of the measurement set-up (quartz bound at the external diameter, convective flows in liquid, presence of bubbles in the liquid, boundary effects in the liquid phase, instability of the reference clock, electric parasitic effects in the front-end electronics, etc.) makes this resolution unachievable. The system was characterized in terms of medium term stability in liquid, and the results are shown in figure 3. The obtained performance is satisfactory, granting a short term measurement resolution of a few Hertz for all up to the fifth harmonic.

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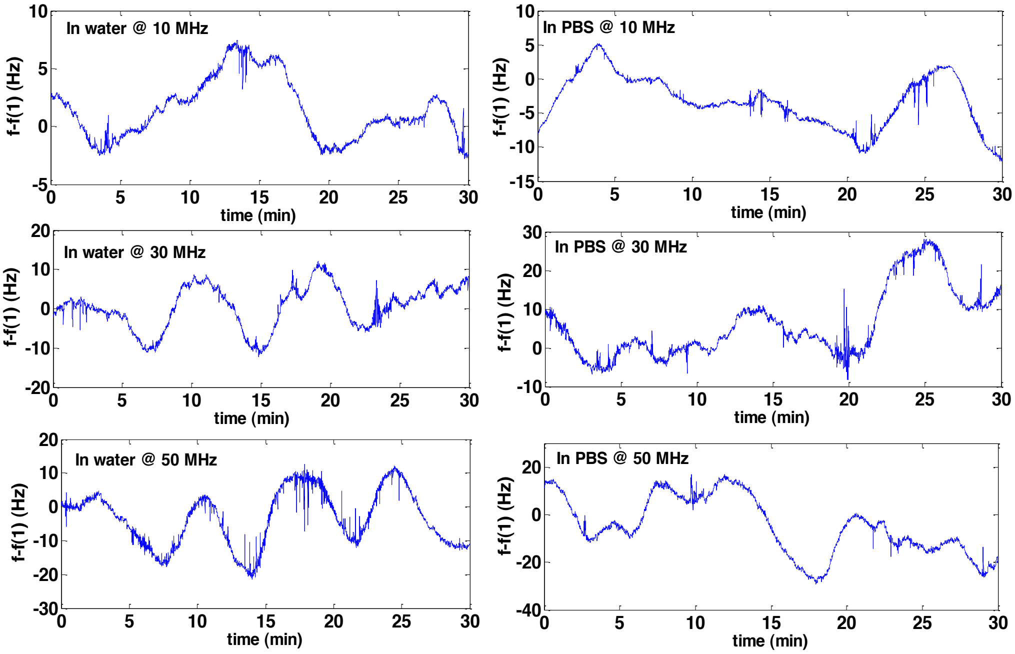

Fig. 3. 1st, 3rd and 5th harmonic: frequency stability in water (left) and PBS (right).