Open Source Quartz Crystal Microbalance with dissipation monitoring

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Abstract. The dissipation factor and subsequently the characterization of the viscoelasticity of deposition films have become crucial for the study of biomolecular adsorption. Most of the commercial quartz crystal microbalance (QCM) systems offer this feature, but it has not been incorporated in open source systems. This article describes the design, construction, and simulation of an open source QCM module for measuring dissipation factor. The module includes two blocks: switch and envelope detector. The switch rapidly disrupts the excitation of the crystal, and connects the output to the envelope detector which demodulates the amplitude of the signal. Damped sinusoidal exponential signals with different time constant were used for simulating viscosity interfaces. The incorporation of few elements facilitated a double-faced PCB design with reduced dimensions. The results from simulation show that the system has a good performance in the range of the biomolecular processes; greater relative error are observed for time constant lower than 1 us. In conclusion, a dissipation module has been developed for calculate dissipation factor using QCM, which is compact and shows great performance for use in biomolecular adsorption.

Keywords: Dissipation, QCM, viscoelastic, envelope.

1. Introduction

Nowadays, new technologies are contributing to facilitate accurate and fast measure of small amount of organic and non-organic substances. The progress of sensor technologies has been feasible due to innovations in different fields including materials and electronic systems. In addition to the development of new tools, applying known technologies to diverse fields has shown to be a great alternative to obtain novel results. In this sense, Open source technologies have shown to have a great impact in the social [1] and the research community [2], due to their easy access and simplicity. Moreover, open source tools rapidly expand to different research fields due to their capability to adapt and satisfy different requirements [3].

In order to determine mass ranging from nanograms to micrograms, several devices has been developed using different technology to measure mass, including cantilever [4], surface acoustic

wave[5], and quartz crystal microbalance (QCM) [6,7]. In particular, QCM is attractive since quartz crystals are massively produced by the electronic industry nowadays.

During last decades, quartz crystal microbalance (QCM) becomes the most utilized mass sensor. QCM is highly sensible to small mass variations due to changes in quartz crystal resonance frequency. Some QCMs reach a resolution up to 0,05ng [8]. QCMs have mayor sensibility than traditional analytical microbalances used nowadays in laboratories. The QCM is based on the piezoelectric effect, when mechanical forces are applied to the sensor producing deformation of the crystalline structure. This results in a dipole moment, and subsequently in currents that can flow across external circuits. On the other hand, applying alternating voltages to the crystal induce mechanical oscillations of the crystal lattice. The resonant frequency of the quartz crystal is established by the distance between the electrodes, and changing the thickness of the resonator (e.g. by thin film mass depositions) results in a shift of the resonant frequency.

Interestingly, the QCM have shown to operate in liquid phase [9], and this feature has been ideal for a vast applications on different fields including chemistry, biology, etc. The real-time quantification of the frequency shifts, due to mass deposition, has facilitated the study of biomolecules absorption or binding such as antibodies[10], proteins[11], and even cells[12]. However, the equations associated with the shift of the resonant frequency and the mass loading assume that the mass must be rigidly adsorbed [13,14]. Therefore, since biological interfaces in the QCM present viscoelastic characteristics, a more accurate index is required. The alternative solution is to measure the energy dissipation due to frictional dissipative losses. This feature often offered in commercial instrument [15] has not been introduced in open source devices.

The present article describes the design, simulation, and fabrication of a dissipation module integrated to a conventional QCM system in order to address dissipation due to viscoelastic film depositions.

2. Materials and methods

2.1. Overview

The dissipation module has been design to be integrated with an open source microbalance system (OpenQCM, http://openqcm.com/). The operating principle of the dissipation module is based on the rate of decay of oscillations generated by a crystal, after a rapid disruption of the excitation voltage. The generated decay voltage can be modeled as an exponential damped sinusoidal, so resonant frequency and dissipation factor can be assessed simultaneously [14,16].

The system consists of a microcontroller interacting to a computer through USB communication (Fig. 1). The system includes two major parts: digital and analog. The digital part not only records the frequency of the input signal, but also commands the switching of the power and the signal, i.e., deactivating the excitation of the crystal and connecting the crystal output to the analog part of the circuit. When signal are connected to the analog circuit, the envelope of the damped sinusoidal is processed by the envelope detector, and then converted to digital by the microcontroller ADC. The switching signal is commanded by the computer.

Figure 1. Block diagram of the QCM system with the dissipation module
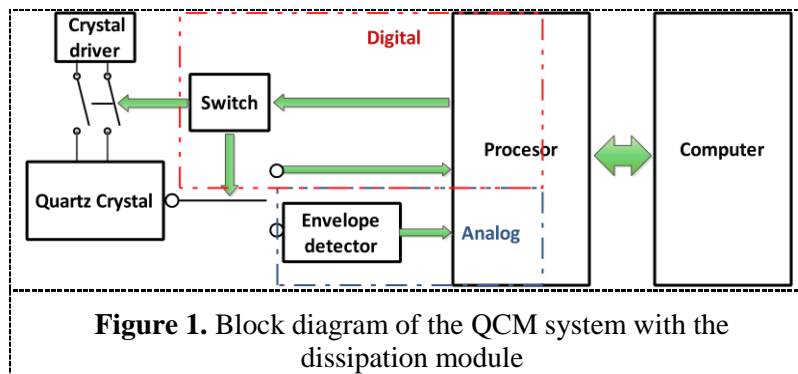
2.2. Hardware

The dissipation module has been adapted to operate with an Arduino micro since it is the core of the original OpenQCM. One GIO pin of the microcontroller control the switching block. The switching is implemented by a digital controlled analog switch using the IC 74HCT4053. When the controlling pin changes the logic level, the switch disconnects the crystal and simultaneously connects the crystal output to the envelope detector circuit. The envelope detector circuit consists of an AM demodulator LTC5507. This IC can operate with carriers ranging from 100 kHz to 1 GHz. The schematic of the system (Fig. 2) was performed in a free PCB designing program (Ealge v7.2, CadSoft).
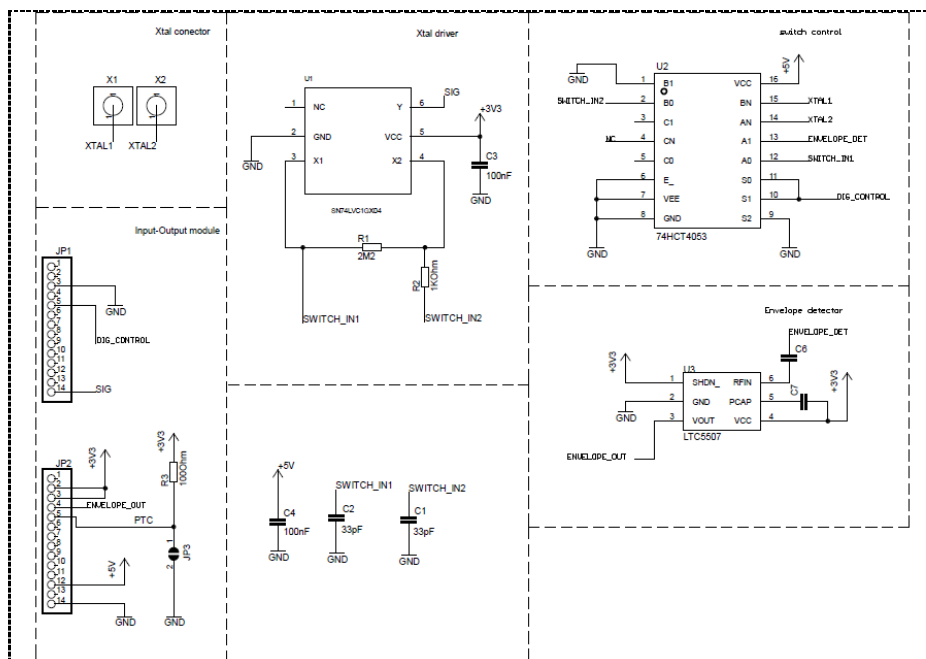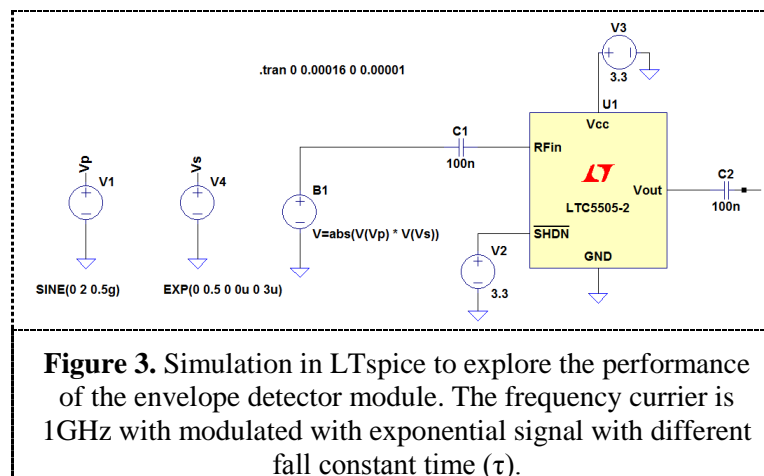

Figure 2. Schematic of the QCM system with the dissipation module. The input-output module facilitates the connection with the arduino board. The switch control deactivates the excitation of the crystal and connects the crystal output to the envelope detector, according to DIG_CONTROL status. The envelope detector quantifies the envelope of the damped sinusoidal and the output is digitalized by the ADC of the microcontroller to estimate the dissipation factor.

2.3. Software

The original user interface to the QCM system has been developed in Java JDK8. The GUI was modified in order to introduce a control switching block button (DIG_CONTROL). In addition, the GUI could be compiled to a standalone executable file.

2.4. Simulation

The performance of the dissipation module was simulated in LTspice. The ltc5505 was used in the simulation, since the ltc5507 is not available in the current version of the simulation program. This IC presents similar operation characteristic with the ltc5507, despites the difference in the frequency working range (100 kHz-1 GHz for the ltc5507 and 300 MHz to 3 GHz for the ltc5505). The sinusoidal damped signals generated by the crystal were simulated using a 1 GHz sinusoidal signal. The currier signal was modulated with different exponentially decreasing function in order to simulate different viscoelastic interfaces; lower time constant (τ) were associated with more viscoelastic interfaces. Tau values used in the simulation were 0.1, 1, 3, 5, and 7 microseconds. The schematic of the simulation is shown in figure 3.

Figure 3. Simulation in LTspice to explore the performance of the envelope detector module. The frequency currier is 1GHz with modulated with exponential signal with different fall constant time (τ).

3. Results

The incorporation of few SMD elements facilitated a double-faced, PTH (plated-through-holes), PCB design with reduced dimensions (Fig. 4). PCB was produced using the ProtoMatS103 PCB milling machine from LPKF. The ProtoMat works straight from CAD data to process boards. This technology provides precise, quick, and simple PCB prototyping, achieving tracks width and clearance of 100 um (Fig. 5). All the components of the OpenQCM and the integration with the designed dissipation module are shown in Fig. 6.
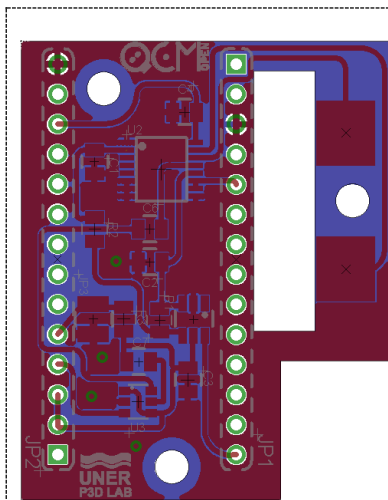

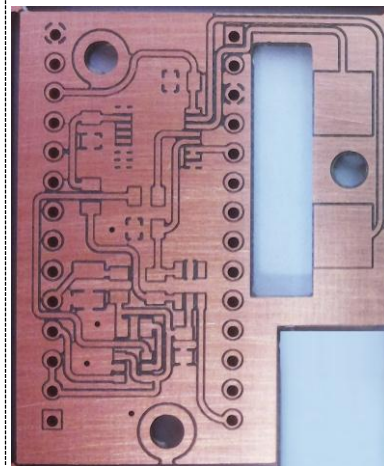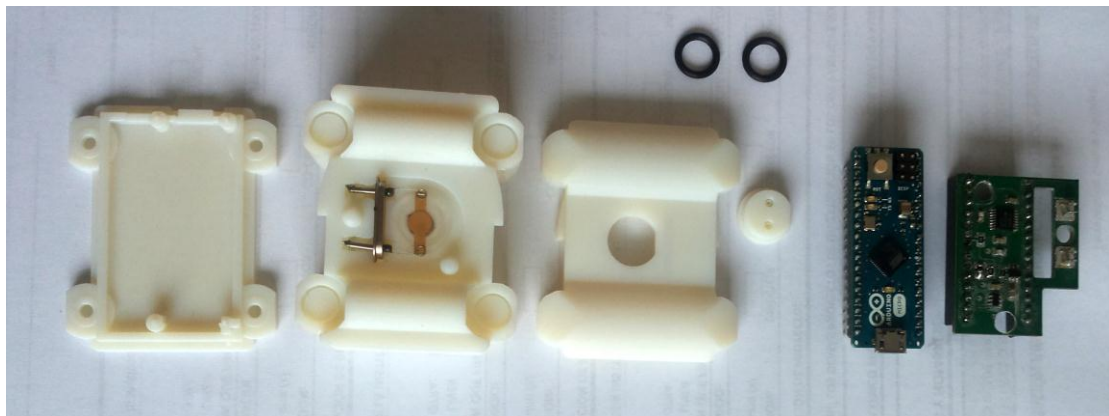|	
Figure 4. PCB design for the QCM with the dissipation module.	**Figure 5.** Board manufactured using a PCB milling system (Protomat, LPKF).

Figure 6. OpenQCM and integration of the dissipation module. The case was printed using a PolyJet 3D printer (Objet Eden260VS®, Stratasys)

The output of the dissipation module due to different input signals is observed in Fig. 7. The response to the selected input of τ with the relative error for each measure is shown in table 1.
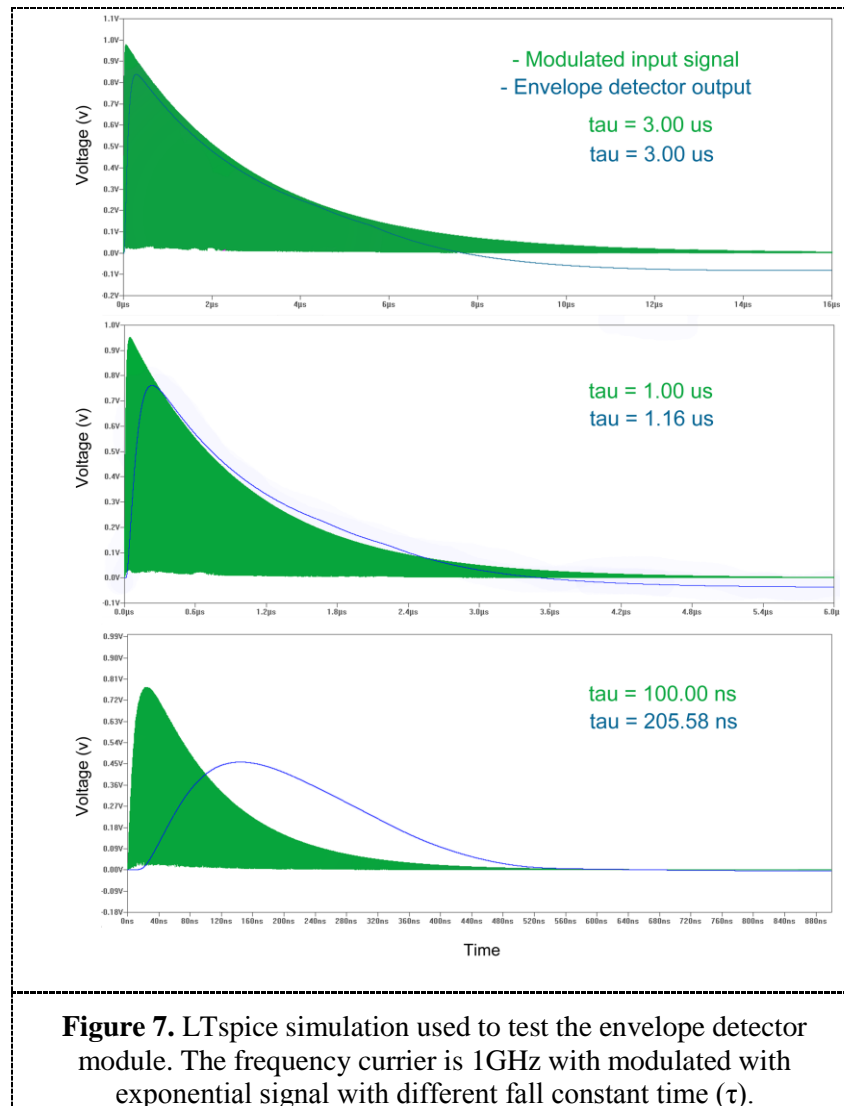
Figure 7. LTspice simulation used to test the envelope detector module. The frequency currier is 1GHz with modulated with exponential signal with different fall constant time (τ).

Table 1. Time constant (τ) estimated (Out τ) at the dissipation module for different exponential damped sinusoidal signal (input τ).

Input τ (us)	Out τ (us)	Error %
0.1	0.2	100
1	1.16	16
3	3	0
5	4.82	3.6
7	7.25	3.57

4. Discussion

The dissipation module is an important enhancement in the open source QCM system. In particular, the reduced number of electronics pieces introduced can be easily incorporated to any system. The present work has described how the module could be successfully integrated to the OpenQCM, showing efficient design.

The quantification of the dissipation phenomenon becomes significant in the study of biomolecular process. For example, during biomolecular adhesion, shifts of frequency without significant dissipation changes are found when the biomolecular mass is rigidly absorbed [14]. Thus, the biomolecules follow the oscillation of the crystal. However, when the absorbed mass is viscoelastic and not hard enough, the biomolecules no longer can follow the oscillation of the crystal, presenting deformation and resulting in energy losses [14]. A previous study showed how two molecules, human serum albumin and antibody from serum albumin, impact differently in the frequency shift and the dissipation factor [13]. The adsorption of the albumin results in shift of the frequency without changes in the dissipation, indicating a rigid characteristic of the serum albumin film. On the other hand, adsorption of antibody yields to large frequency and dissipation changes, reflecting the viscoelasticity characteristic of the biomolecular film.

Interestingly, the simulation shows that the dissipation module has low relative error for modulated exponential signals with time constant higher than 1 us. The time constant is inversely proportional to the dissipation factor ($D = 1 / f\pi\tau$) [14]. The typical time constant decay for a 10 MHz QCM in air has estimated to be around 2-3 ms. Likewise, the QCM with deionized water presents a decay constant time around $x10^{-3}$s, and around $x10^{-4}$s for proteins such as protein A and fibronectin [17]. Most of the dissipation factor for biomolecular adsorption are in the range of single to tens ($\times 10-6$) of dissipation units [13]. Therefore, the present module is able to characterize biomolecular viscoelastic films, because it is capable of measuring faster decay time constant. This feature could be used for characterization of cell adhesion on the surface of implants, which is critical to assess biocompatibility (although additional investigation are required to reduce the size of the sensor), or it can be include into label-free sensors.

It has to be mention that most of the commercially available peak voltage detector, including the LTC5507, presents non-linearity for small signals [18]. This effect can be noted at the end of the decay exponential in Fig 7, although low-level voltages are not used for quantifying the time constant decay in the case of the QCM. Nevertheless, this non-linearity observed in commercial peak-voltage detector IC has been associated with diode performance, and can be sort out by using circuits without diodes or other nonlinear components, enhancing significantly the linearity of the system [19]. In addition, further analysis is needed in order to calculate the resolution of the system. The resolution to estimate the dissipation factor can be increased by fitting an exponentially damped sinusoidal. Thus, works in this direction are being developed by the authors.

In conclusion, a dissipation module has been developed for quantifying dissipation factor using QCM. The system is compact and can be easily incorporated to any QCM system. In addition, the module exhibit satisfactory performance for measuring viscoelasticity on the interface, addressing on real-time the quantification of time decay constants usually observed in biomedical applications. In particular, the module results a desirable system for studying biomolecular adsorption.

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