QCM Sensor System Based on a Phase Detector Circuit for Measurements of Density and Viscosity of Liquids

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Abstract— **In this work a QCM sensor system based on the phase shift detection method is implemented. The system is characterized for liquid measurement applications. The experimental calibration of the sensor system is performed with solutions of increasing viscosities, obtaining the equation of the sensitivity of the output voltage to the changes in the square root of the density-viscosity product of the liquid.**

Keywords— QCM sensor; phase detection; liquid medium, sensitivity; density; viscosity

I. INTRODUCTION

The AT-cut quartz crystal resonator is frequently used as a sensor in a large number of applications due to its good stability against temperature changes. In an AT-cut quartz crystal resonator the maximum displacement for the different modes of resonance occurs on the surface of the crystal, which makes the device sensitive to surface perturbations. If mass is deposited on the surface of the resonator, there is an alteration of the resonance frequency proportional to the amount of added mass [1], having a QCM (Quartz Crystal Microbalance) sensor. In 1985, Kanazawa and Gordon [2] developed a simple model that describes the operation of a QCM resonator in liquid medium. Later, using a continuous electromechanical model, Martín and Granstaff obtained in 1991 the expression of the electrical admittance of the quartz-mass-liquid group as a function of the excitation frequency [3].

The presence of fluid and deposition alter the series resonance frequency. The use of the quartz resonator in the feedback network of an electronic oscillator circuit allows the changes in the resonance frequency to be determined from variations in the oscillation frequency of the circuit. The low cost of the oscillators and the possibility of "continuous" monitoring in real time make the oscillators a good choice for most QCM sensor applications. However, variations in the oscillation phase produce instabilities of the oscillation frequency decreasing the resolution of the sensor [4]. In order to reduce frequency fluctuations, Arnau et al. [5] have proposed a new measurement technique. This new technique involves interrogating the crystal at a very stable fixed frequency and measuring changes in the phase of the QCM resonator. In [6], the authors have experimentally verified that the phase variation is proportional to the variation of the mass surface density at the resonator electrode, so they say this method allows the development of new QCM sensor systems that are alternative to the oscillators.

In this work, the calibration of a QCM sensor system based on a phase detection circuit was carried out in order to characterize the properties of a liquid. Initially the electronic system implemented is described, based on that described by Montagut et al. in [6].

II. DESIGN OF THE SENSOR SYSTEM

The design of the sensor system was made following [6]. Fig. 1 shows the block diagram of the system proposed by Montangut et al. Fig.2 shows the implemented sensor system. As a QCM sensor, a 9MHz crystal from Inficon was used. The IC AD9913 Direct Digital Synthesizer (DDS) from Analog Devices was chosen to generate the test frequency. The AD9913/PCBZ Evaluation Board was used with an external 25MHz crystal as device clock oscillator and a PLL 2x multiplier to obtain a frequency around crystal resonance (9MHz). Its short-term stability was determined by calculating the Allan deviation [7]. A maximum noise $\sigma_{max} = 2{,}56.10^{-8}$ was found for an integration time of 1s ($\sigma_{max} f_0 = 0{,}23 Hz$). The IC AD8302 from Analog Devices was used as phase detector, the same one used by Montagut et al [6]. Before implementing the system, the relationship between the phase shift at the

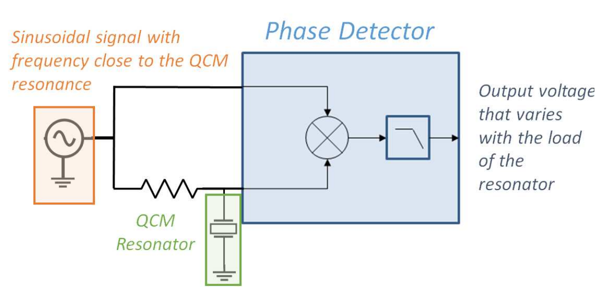

Fig. 1. Block diagram of the sensor system based on the work of Montangut et al. [6].

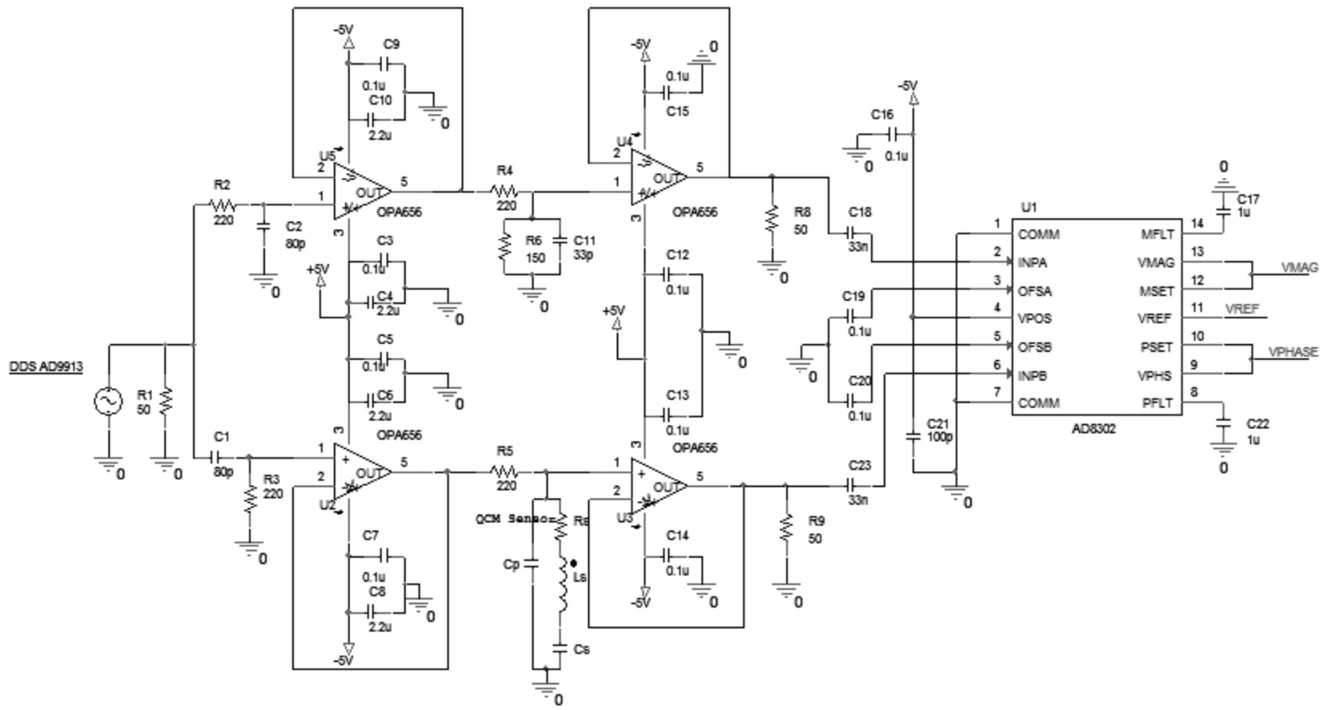

Fig. 2. Schematic of the sensor system.

detector inputs and the output voltage was experimentally determined:

$$V_{phase} = -0{,}0106 \mid \Delta\varphi \mid +1{,}792 \; (0^o<|\Delta\varphi|<140^o). \quad (1)$$

In the branches connected to the phase detector inputs (QCM branch and R_5-C_{11} branch that simulates the behavior of the QCM resonator in water), the voltage buffers were implemented using low-noise operational amplifiers OPA656 from Texas Instrument.

III. Characterization of the Sensor System for Liquid Measurements. Calibration and Sensitivity Equation

To study the sensitivity of the sensor system to changes in the square root of the product density-viscosity of a liquid, solutions of glycerol in water of different concentrations were used (Table I). The test frequency (f_t = 9.0000044 MHz) was chosen by studying the frequency response of the system in a resonance environment, with the QCM sensor submerged in each mixture (see Fig. 3). Subsequently, at a fixed frequency f_t, the output voltage (V_{phase}) was monitored as a function of time, with a sampling interval of 1s for 5 min per dissolution, and the sensitivity curve to changes in $\Delta\sqrt{\rho\eta}$ was determined. In Fig. 4 this curve is shown. The sensitivity equation for the sensor system was calculated. Results are indicated in (3), where A_1, A_2 and A_3 are the sensitivity coefficients measured at the reference solution $\sqrt{\rho\eta}_0 = 1{,}0533\sqrt{\boldsymbol{g/cm^3 \cdot cp}}$ and V_0=0,6657V is the output voltage at $\sqrt{\rho\eta}_0$.

$$\frac{\Delta V[V]}{V_0} = A_3\left(\Delta\sqrt{\rho\eta}\right)^3 + A_2(\Delta\sqrt{\rho\eta})^2 + A_1\left(\Delta\sqrt{\rho\eta}\right) \quad (3)$$

TABLE I. Density and viscosity of glycerol/water mixtures

% glycerol	ρ (25ºC) (g/cm^3)	η (25ºC) (cp)
8	1,01599	1,0920
21	1,04846	1,5920
39	1,09444	3,0520
46	1,11309	4,1650
51	1,12649	5,3190
59	1,14835	8,3223
63	1,15927	10,8177
66	1,16746	13,2329
72	1,18381	21,2402
78	1,20004	37,1449

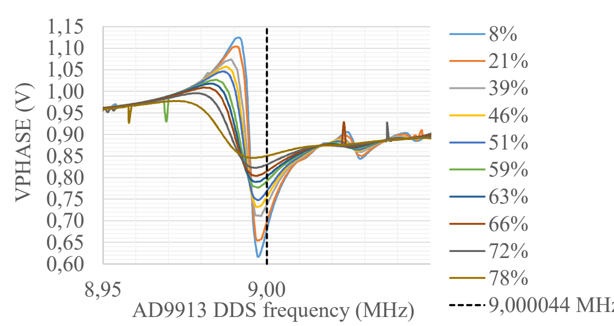

Fig. 3. Output voltage as a function of the DDS frequency for each glycerol/water mixture. Selected excitation frequency: 9.000044MHz.

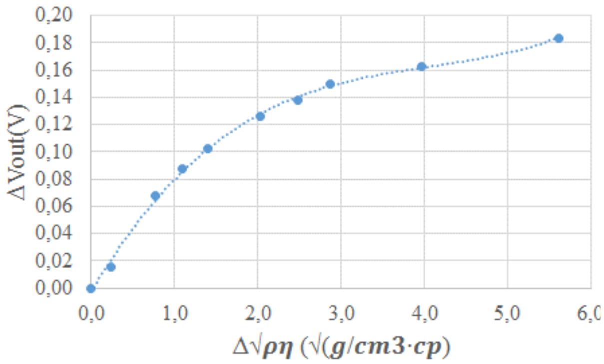

Fig. 4. Variation of the output voltage of the sensor system versus the variation of the square root of the density-viscosity product of the liquid. Sensitivity curve.

where $A_1 = 1{,}36 \cdot 10^{-1} / \left(\sqrt{\boldsymbol{g/cm^3 \cdot cp}}\right)$

$$A_2 = -3{,}31 \cdot 10^{-2} / \left(\sqrt{\boldsymbol{g/cm^3 \cdot cp}}\right)^2$$

$$A_3 = 2{,}81 \cdot 10^{-3} / \left(\sqrt{\boldsymbol{g/cm^3 \cdot cp}}\right)^3$$

Finally, in order to validate the operation of the system, the output voltage was converted into phase shift using the experimental equation obtained for the IC AD8302. Using the equivalent electrical model of the QCM sensor loaded with the liquid [3], the expected theoretical phase shift was determined for each mixture (see Fig. 5), checking that the sensor system works properly.

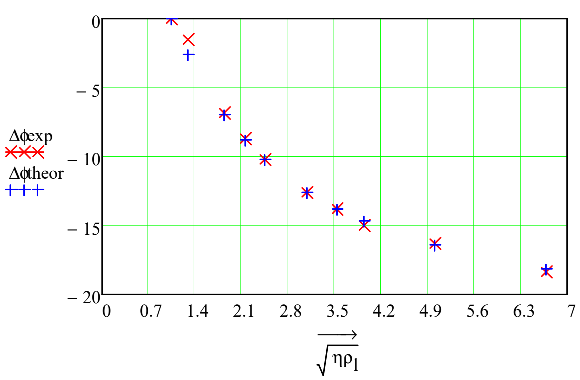

Fig. 5. Phase shift versus $\sqrt{\rho\eta}$. Comparison between theoretical and experimental results.

IV. Summary and Conclusions

In this work, a sensor system based on the phase detection method described by Arnau and Montagut in the literature [5][6] has been implemented and characterized. To generate the excitation signal at the input of the sensor system, the direct digital tuner circuit (DDS) AD9913 was used. The frequency noise of the DDS was characterized using the Allan deviation, finding that, for measurements with a sampling period of one second, the frequency deviation for test signals in an environment of 9MHz is 0.23Hz. The AD8302 phase detector circuit used was experimentally characterized, determining the relationship between the variation of the output voltage and the phase shift between its inputs. Subsequently, the operation of the QCM sensor system for liquid measurements was characterized using glycerol solutions in water of different concentrations. The optimum test frequency for the application was determined. Experimental calibration of the sensor system was performed, obtaining the equation of sensitivity of the output voltage to the changes in the square root of the density-viscosity product of the liquid.

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