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# FULL SYSTEM FOR PIEZOELECTRIC ORGANIC MEMS RESONATORS OPERATING IN LIQUID MEDIA

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## ABSTRACT

Here is presented a full compact system enabling the integrated actuation and measurement of the resonance frequency of piezoelectric organic MEMS resonators in liquid media. The resonators contain a piezoelectric layer made of PVDF-TrFE for both the actuation and the read-out of the MEMS. They are included into a watertight enclosure associated with a PCB and a dedicated electronic card, allowing electrical measurements in air and liquid media. An application for viscosity measurements of water/glycerol mixtures is presented and shows that results match with measurements made with a viscometer, without any knowledge of the mass density.

## KEYWORDS

Organic Piezoelectric MEMS, Measuring System, Liquid Media, Resonance Frequency, Viscosity.

## INTRODUCTION

The success and development of MicroElectroMechanical Systems (MEMS) does not need to be demonstrated since they are ubiquitous in every technology. A recent trend goes towards the use of organic materials in MEMS [1]. Despite the fact that processing technologies are not as mature as for silicon, organic electronic materials offer other possibilities and are really promising for future applications. Indeed, organic materials are much more flexible and attractive for all flexible and biomedical applications [2]. Moreover, organic materials properties are easily tunable, which enables to conceive new applications such as in [3], and the manufacturing costs are also much lower.

In the present work, organic piezoelectric MEMS resonators are developed and used in liquid media as viscosity sensors. In order to have a good read-out, all the measurement chain has to be taken into account (transduction, signal conditioning and treatment). That is why it is important to have an efficient measuring system, especially for resonators operating in liquid media where damping limits the application of such resonators. In this context, here is presented a full system that enables electronic measurements of the resonance frequency of organic piezoelectric resonators in liquid media.

## MEASUREMENT STRATEGY

The fabrication process of the resonators used in this work is detailed in [4]. The resonators are composed of a poly(ethylene naphthalate) (PEN) substrate, a piezoelectric polymer made of poly(vinylidene fluoride-trifluoroethylene) (PVDF-TrFE) lying between two aluminum electrodes, and of poly(dimethylsiloxane) (PDMS) that covers all the cantilever for the passivation of the structures (Fig. 1). The patterning of the cantilevers shape is obtained simply by xurography.

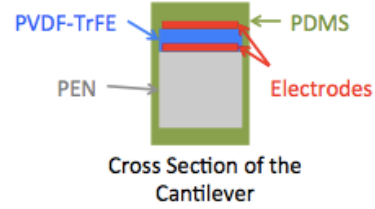


Figure 1: Architecture of the organic piezoelectric MEMS resonators.

In order to determine the resonance frequency of the fabricated resonators, it has been chosen to measure the admittance spectrum. From an electrical point of view, a piezoelectric resonator can be modeled as presented in Fig. 2. The motional part of the cantilever is represented as a RLC circuit. Due to the architecture of the resonators, a static capacitance  $C_0$  and a resistance  $R_0$  are parallel to the motional branch.

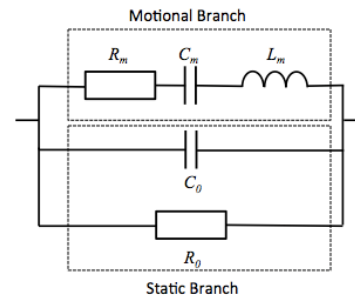


Figure 2: Electrical model of the piezoelectric resonators.

Actually, in order to measure the resonance frequency, only the motional branch must be taken into account, as  $C_0$  and  $R_0$  do not participate in the motion of the cantilever. Indeed, Fig. 3 shows the measurement of both the real part (conductance  $G$ ) and the imaginary part (susceptance  $B$ ) of the total admittance. A positive slope is visible for both the conductance and the susceptance. Actually, the relative dielectric permittivity of the organic piezoelectric material is a complex number and can be expressed as:

$$\epsilon_r = \epsilon_r' - j\epsilon_r'' \quad \text{with} \quad \epsilon_r' > 0 \quad \text{and} \quad \epsilon_r'' > 0 \quad (1)$$

Moreover, the admittance of the static branch can be expressed as:

$$Y_s = j \frac{\epsilon_0 \epsilon_r S}{h_p} \omega = j \frac{\epsilon_0 \epsilon_r' S}{h_p} \omega + \frac{\epsilon_0 \epsilon_r'' S}{h_p} \omega = jC_0 \omega + \frac{1}{R_0} \quad (2)$$

with  $h_p$  the PDF-TrFE thickness,  $S$  the electrode surface,  $\epsilon_0$  the vacuum permittivity and  $\omega$  the angular frequency. Thus, both the real and the imaginary parts of  $Y_s$  are proportional to the frequency, which explains the slopes for both  $G$  and  $B$ . Since the resonance frequency corresponds to the maximal value of the conductance without the effect of  $R_0$ , this effect must be canceled. It is

possible to cancel it numerically as shown in Fig. 3, with a code that calculates the coupling in order to reduce the data to a second order band-pass system [5]. Using this method, the coupling has been determined and subtracted, giving the admittance spectra presented in Fig. 4. Without the coupling, the resonance frequency corresponds to the maximum of the conductance. This method is efficient but it is necessary to process the data to cancel the coupling. However, the determination of the equivalent electrical model is possible with these measurements: without the coupling,  $G_{max} = 1/R_m$ . Moreover, the quality factor enables to calculate  $C_m$  and  $L_m$  since  $Q = L_m\omega_0/R_m$  and  $\omega_0^2 = 1/(L_m C_m)$ . Finally,  $C_0$  and  $R_0$  can be calculated taking the value of the coupling at  $f_0$  for  $B$  and  $G$  respectively. The following values are calculated:  $R_m = 4.76 \text{ M}\Omega$ ,  $L_m = 393 \text{ H}$ ,  $C_m = 26.4 \text{ fF}$ ,  $R_0 = 440 \text{ k}\Omega$  and  $C_0 = 80.9 \text{ pF}$ . Afterwards, the equivalent model is represented in Fig. 3 and 4 using these values. There is a good correlation between measurements and electrical modeling around the resonance frequency. Using the determined values of  $R_0$  and  $C_0$  and Eq. 2, the real and imaginary parts of the relative dielectric permittivity are calculated:  $\epsilon'_r = 7.6$  and  $\epsilon''_r = 0.69$ .

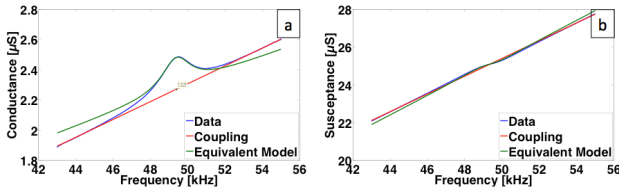


Figure 3: Measurement of a) the real part (conductance) and b) the imaginary part (susceptance) of the total admittance of the resonators around a resonance frequency.

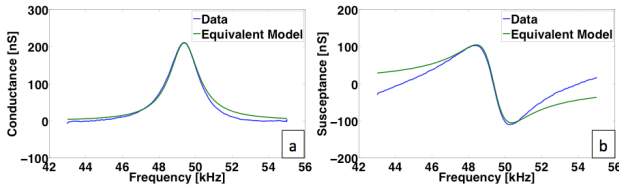


Figure 4: a) Conductance spectrum and b) susceptance spectrum without the coupling due to both  $R_0$  and  $C_0$ .

In order to avoid this post-measurement process, the strategy is to electronically cancel the effect of both  $C_0$  and  $R_0$ . With this in mind, a differential measurement between the cantilever that is free to move and a reference one that is unreleased (mechanically stuck) is performed. Indeed, out of the resonance, if the two cantilevers have the same geometry and materials (i.e. same  $C_0$  and  $R_0$ ), the effect of the static branch is canceled with a differential measurement. Nevertheless, around the resonance frequency, the behavior of the cantilever that is free to move differs from the fixed one, which enables the differential electrical measurement to only depend on the signal of the motional branch. With the aim of having the same structure for the free cantilever and the unreleased one, both of them are fabricated at the same time on the same substrate. Figure 5 shows the structure of the

resonators together with the pattern of the electrodes in order to have the same geometry for the two cantilevers.

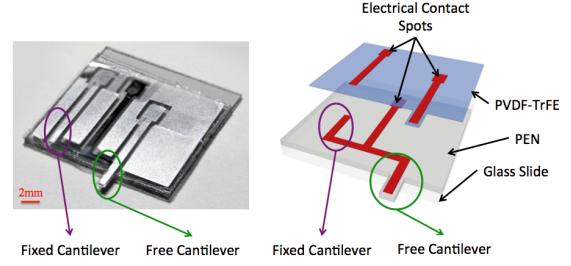


Figure 5: Picture of a resonator with identification of the free cantilever and the fixed one, as well as an exploded view of the structure in order to have a better understanding.

## VNA CARD

In order to implement the strategy developed previously for electrical measurements, a dedicated electronic card has been designed. The working principle of this card is basically the same as a Vectorial Network Analyzer (VNA). This card enables both the actuation and the read-out of the resonance spectrum. Figure 6 presents a synopsis of the card and the measurement principle.

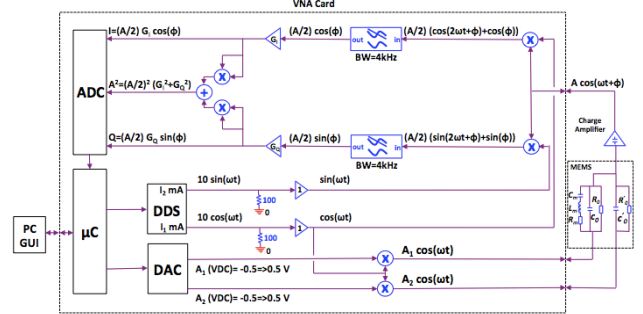


Figure 6: Synopsis of the VNA card and of the measurement principle.

The two cantilevers (fixed and released ones) are powered with two synchronized sinusoidal voltages whose amplitudes can be set separately. The signal coming from the common electrode goes to a charge amplifier and then, the principle of an IQ demodulator is applied. One part of the signal is multiplied by an in-phase signal when another part is multiplied by a quadrature signal. This gives two signals with a component at twice the starting frequency and a continuous component. In order to only keep the continuous component, these signals go to a low-pass filter. Finally, with the two continuous signals, it is possible to determine the modulus  $A$  and the phase  $\phi$  of the signal that comes out of the charge amplifier. Homemade software is used to set the different parameters and to acquire and visualize the spectra.

Normally, if the two cantilevers are exactly the same and powered with two voltages of opposite phase and same amplitude, the output signal of the charge amplifier is independent of  $R_0$  and  $C_0$  and it is proportional to  $Y_m/\omega$  (with  $Y_m$  the admittance of the motional branch). However, the two cantilevers are never exactly identical and thus the compensation is not optimal. Figure 7 illustrates three different configurations. Firstly, if the

measurements are done without the compensation of  $R_0$  and  $C_0$ , the resonance is hardly visible (Fig. 7a). Secondly, with a compensation using the same amplitude for the two powering voltages, the resonance is more obvious but it still does not look like an ideal spectrum with a peak for the modulus and a  $180^\circ$  shift for the phase, which means that the compensation is not optimal (Fig. 7b). Finally, it is possible to adjust the amplitudes of the two voltage sources in order to electrically compensate what is not physically compensated. Thus, it is possible to obtain usable spectra that make the determination of the resonance frequency possible as it corresponds to the maximum value of the modulus (Fig. 7c). The strategy proposed here with the electronic card is efficient and enables to obtain good quality resonance spectra.

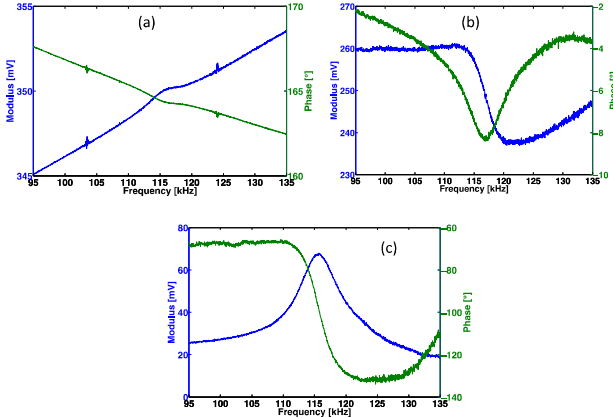


Figure 7: Modulus and phase spectra in air for three different configurations: a) without any compensation, b) with a compensation with the same amplitudes, c) with a compensation with tuned amplitudes.

## WATERTIGHT ENCLOSURE

It has been shown that the electronic system is efficient and enables effective measurements of the resonance spectrum. However, the results that are presented correspond to resonators operating in air. In this work, the resonators are used in liquid media. That is why a system that makes the measurements in liquid media possible is required. For this purpose, a watertight enclosure has been fabricated and presented in Fig. 8.

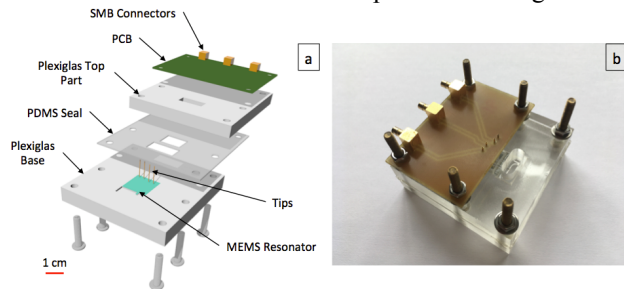


Figure 8: a) Exploded view of the watertight enclosure, b) picture of the system.

The aim of this system is to separate the cantilever operating in the liquid media from the electrical contacts that must stay dry, especially for conductive liquids. The watertight enclosure is composed of two Plexiglas parts sandwiching a seal made of PDMS (Sylgard 184). To transfer the electrical contacts of the devices, spring tips are used. Four tips are used: one for the bottom electrode,

two for the top electrodes and a last one for the ground plan. These tips pass through the top Plexiglas part and are soldered to a Printed Circuit Board (PCB) that comes on top of the watertight enclosure. Finally, the PCB is connected to the VNA card via SMB connectors. Thanks to this system and the VNA card, measurements in liquid media are possible. An example is given in Fig. 9, with a comparison between air and water measurements. For better understanding of this complete measuring system, a summary is presented in Fig. 10.

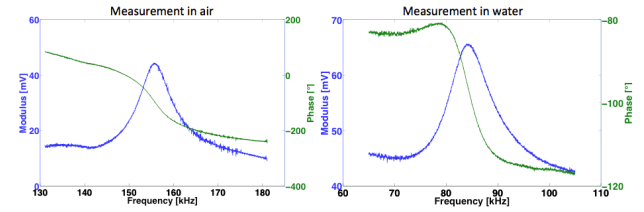


Figure 9: Example of measurements of the resonance frequency in air and in water for the same resonator.

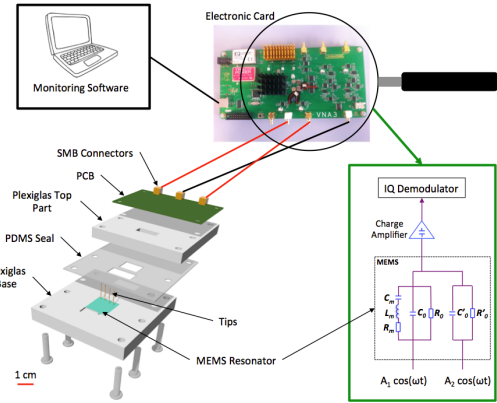


Figure 10: Summary of the full measuring system.

## VISCOSITY MEASUREMENT

In this work, the organic resonators are used as viscosity sensors in liquid media. Theoretically, it is possible to determine both the viscosity and the mass density of a fluid by measuring both the resonance frequency  $f_r$  and the quality factor  $Q$  of the MEMS resonators. In this case, six parameters ( $m_1$ ,  $m_2$ ,  $m_3$ ,  $c_1$ ,  $c_2$  and  $c_3$ ) must be calculated [6]:

$$1/(2\pi f_r)^2 = m_1 + m_2\rho + m_3\sqrt{\eta\rho}/(2\pi f_r) \quad (3)$$

$$1/Q = 2\pi f_r (c_1 + c_2\eta + c_3\sqrt{2\pi f_r\eta\rho}) \quad (4)$$

with  $\eta$  the viscosity and  $\rho$  the mass density of the fluid. It means that a calibration with three liquids whose mass densities and viscosities are known is necessary to determine these coefficients. Then, the viscosity and the mass density can be analytically and independently determined for random liquids [7]:

$$\rho = \frac{1}{2}(a_0k_1 - b_0 + k_3) \frac{m_3}{m_2k_2\sqrt{2\pi f_r}} \quad (5)$$

$$\eta = \frac{1}{2}(b_0k_1 - a_0 + k_3) \frac{c_3\sqrt{2\pi f_r}}{c_2k_2} \quad (6)$$

with:



$$a_0 = \frac{1}{m_3(2\pi f_r)^{3/2}} - \frac{m_1}{m_3} \sqrt{2\pi f_r}, \quad b_0 = \frac{1}{c_3 Q(2\pi f_r)^{3/2}} - \frac{c_1}{c_3 \sqrt{2\pi f_r}}$$

$$k_1 = 1 - 2 \frac{m_2 c_2}{m_3 c_3}, \quad k_2 = 1 - \frac{m_2 c_2}{m_3 c_3}, \quad k_3 = \sqrt{(a_0 - b_0)^2 + 4a_0 b_0} \frac{m_2 c_2}{m_3 c_3}$$

Concretely, measurements of the resonance frequency and the quality factor have been made in water/glycerol mixtures for different concentrations of glycerol in order to determine their viscosity. In a first set of experiments, the dynamic measurements are made with a laser Doppler vibrometer for the first flexural mode of the piezoelectric cantilevers and the results are presented in Fig. 11.

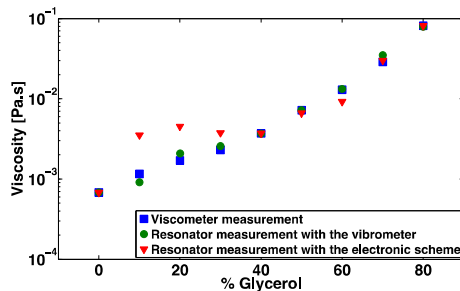


Figure 11: Viscosity determinations with two methods: one using a commercial viscometer and the other one using the resonance frequency of two modes of the resonators.

The viscosities determined with the resonators are compared with the ones measured with a viscometer. The results with the resonators are consistent with the viscometer, with a maximum relative error of 22 %, and prove the efficiency of organic resonators for measurements in liquid media. Moreover, there is no need of the mass density value to determine the viscosity, contrary to commercial viscometers. Afterwards, measurements have been made with the electronic scheme but for a resonance mode that is a combination of longitudinal and lateral flexions, as it is the one that presents the best electrical response. However, due to the electrical coupling that is not entirely compensated, the measurements of the quality factor are misrepresented. That is why a post-measurement process has been used to cancel the remaining coupling and to calculate the quality factor. Thus, the viscosity has been calculated and the results are also consistent (Fig. 11), with a maximum relative error of 32 %, except for the smallest viscosity values. Similar measurements are carried out with inorganic cantilevers, where the authors use the resonance frequency and the quality factor of two different modes to determine both the mass density and the viscosity with an error around 3 % [8]. In another article, the authors only use the resonance frequency of two modes in air and in water, and then in the liquid of interest in order to calculate the mass density and the viscosity with an error around 8 % [9].

## CONCLUSIONS

In this work, a full compact measuring system for organic piezoelectric MEMS resonators operating in liquid media is presented. The VNA card enables to

measure the resonance spectrum of the resonators and to compensate the static branch. The VNA card is associated with a watertight enclosure in order to use the resonators in liquid media. Finally, an application for the determination of fluids viscosity is carried out. Measuring the resonance frequency and the quality factor in the fluid enables to calculate the viscosity without any knowledge of the mass density. The results are in a good agreement with the measurements done with a commercial viscometer for a large viscosity range (two decades). This work is promising for future biological and/or chemical applications in liquid media.

## ACKNOWLEDGEMENTS

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