Sensors and Actuators B 254 (2018) 291–298

Contents lists available at ScienceDirect

Sensors and Actuators B: Chemical

journal homepage: www.elsevier.com/locate/snb

Flow-through sensor based on piezoelectric MEMS resonator for the in-line monitoring of wine fermentation

J. Toledo [a,*], V. Ruiz-Díez [a], G. Pfusterschmied [b], U. Schmid [b], J.L. Sánchez-Rojas [a]

[a] *Microsystems, Actuators and Sensors Group, Universidad de Castilla-La Mancha, Ciudad Real, Spain*
[b] *Institute of Sensor and Actuator Systems, TU Wien, Vienna, Austria*

ARTICLE INFO

Article history:
Received 18 January 2017
Received in revised form 13 July 2017
Accepted 14 July 2017
Available online 17 July 2017

Keywords:
Microresonators
Piezoelectric
Oscillator circuit
Density
In-line
Grape must fermentation

ABSTRACT

The traditional procedure followed by winemakers for monitoring grape must fermentation is not automated, has not enough accuracy or has only been tested in discrete must samples. In order to contribute to the automation and improvement of the wine fermentation process, we have designed an AlN-based piezoelectric microresonator, serving as a density sensor, resonantly excited in the 4th-order roof tile-shaped vibration mode. Furthermore, conditioning circuits were designed to convert the one-port impedance of the resonator into a resonant two-port transfer function. This allowed us to design a *Phase Locked Loop*-based oscillator circuit, implemented with a commercial lock-in amplifier with an oscillation frequency determined by the resonance mode. We measured the fermentation kinetics by simultaneously tracking the resonance frequency and the quality factor of the microresonator. The device was first calibrated with an artificial model solution of grape must and then applied for the in-line monitoring of real grape must fermentation. Our results demonstrate the high potential of MEMS resonators to detect the decrease in sugar and the increase in ethanol concentrations during the grape must fermentation with a resolution of 1 mg/ml and 20 µPa s as upper limits for the density and viscosity, respectively.

1. Introduction

The fermentation of grape must involves the interaction between yeasts, bacteria, fungi and viruses. A correct bio-chemical process is a necessary condition but not sufficient by itself to determine the final quality of the wine, whose assessment relies on a comprehensive analysis of its chemical components, as the basic flavour of wine depends on at least 20 compounds [1]. Therefore, winemakers must carefully supervise the wine fermentation process to ensure a wine of the expected quality. One of the key parameters monitored during wine fermentation is the fermentation kinetics. This provides essential information about the steady transformation of grape must into wine due to the decrease of glucose and fructose that leads to the formation of ethanol, glycerol and carbon dioxide along with biomass, as a result of yeast metabolism [2]. This process is traditionally monitored by enologists, who manually extract and analyse discrete samples at least twice a day using instruments such as the aerometer, spectrophotometer or colorimeter. They essentially determine the density and its rate of change since these parameters provide information about the evolution of the fermentation as a result of yeast metabolism. In an industrial fermentation process, temperature fluctuations would influence to some extent the fermentation process and thus the density. Nevertheless, it is the variations that help enologists determine the current fermentation status, and whether or not corrective measures are required as it progresses.

Besides, a variety of alternative procedures have been used for monitoring the grape must fermentation such as density determination based either on differential pressure measurements [3] or on the use of flexural oscillators [4], monitoring of CO_2 released during the process due to the gradual loss of mass [3,5], determination of yeast cell population evolution by means of impedance techniques [6] and turbidity measurements [7], ultrasound measurements conducted to determine the propagation velocity in grape musts [8,9], refractive techniques based on fibre optics [10], optoelectronic device based on measurements of the refractive index [11,12] and an off-line monitoring based on a piezoelectric resonator [13].

Overall, these different approaches have not enough resolution or have only been tested in discrete must samples. In this context, the simultaneous determination of the density and viscosity of liquids, through measurement of the resonant frequency (f_r) and quality factor (*Q-factor*) of a mechanical resonator, has already been reported [14–16]. This approach presents several advantages with

---

* Corresponding author.
E-mail address: javier.toledo.serrano@gmail.com (J. Toledo).

http://dx.doi.org/10.1016/j.snb.2017.07.096
0925-4005/

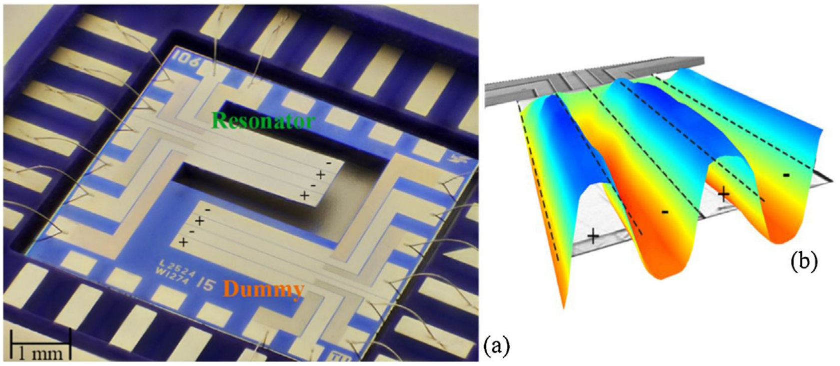

Fig. 1. (a) Top-view micrograph of the resonator. (b) Modal shape measured (*15-mode*) with a laser Doppler vibrometer.

respect to traditional methods, such as real-time analysis, on-line configuration and low liquid volumes. Piezoelectric resonators with in-plane vibration modes [17], flexural and torsional modes [18] have already been tested. However, it is well-established that a different family, such as the roof tile-shape modes, present better quality factors at moderate frequencies [19,20]. The present work evaluates the performance of this vibration mode, within micromachined self-actuated and self-sensing aluminium nitride (*AlN*)-based cantilever sensor for the in-line monitoring of grape must fermentation in flow-through configuration.

2. Material and methods

2.1. Resonator design and characterization

The resonator was designed for the 4th order roof-tile shaped vibration mode. It resulted in a cantilever resonator with optimized electrode layout featuring five nodal lines in one direction, and 1 nodal line in the perpendicular direction. Considering Leissa's nomenclature, the vibration mode is named as *15-mode* [21]. The resonator was designed with a length of L = 2524 μm, width of W = 1274 μm and thickness T of 20 μm. It was fabricated from a SOI wafer with a 20 μm-thick device layer covered with a 650 nm-thin AlN piezoelectric film [22]. The top metallization has four striped electrodes that allow a selective excitation of the vibration modes and act as a filter for higher modes [18]. This device has already been tested in a previous work obtaining a quality factor of 116 in 2-Propanol [23]. The considered modal shape and the fabricated resonator are shown in Fig. 1 [13].

2.2. PLL-based oscillator

The simultaneous determination of density and viscosity of liquids, through measurement of the resonant frequency and the quality factor of a mechanical resonator, is challenging due to the low quality factors and parasitic effects present in liquid media [24]. For this reason, our approach is based on roof tile-shaped resonators in two-port configuration and a parasitic compensating device.

In the two-port scheme, one of the electrodes was used for actuation (+) and the other for sensing (−) (see Fig. 1). However, our results show that a capacitive crosstalk (C_{ft}) across the actuation and sensing ports has a significant contribution to the output current. To minimize this parasitic component, a compensating device was introduced. This dummy device reproduces the structure of the resonator, but without the backside release, thus preventing any vibration. For this reason, we designed an interface circuit, used in a previous work [16], to subtract the dummy response from that of the resonator. Since the materials and dimensions in the dummy and resonator devices are the same, they are expected to show identical electrical behaviour with respect to their parasitic effects. This results in a clear resonance, with low baseline (see Fig. 2) and remarkable phase transition [13]. However, a phase shift (about −12°) is introduced by the instrumentation amplifier (Fig. 2) at the maximum gain, representing the differences in electrical performance between the dummy, the resonator device and C_{ft}.

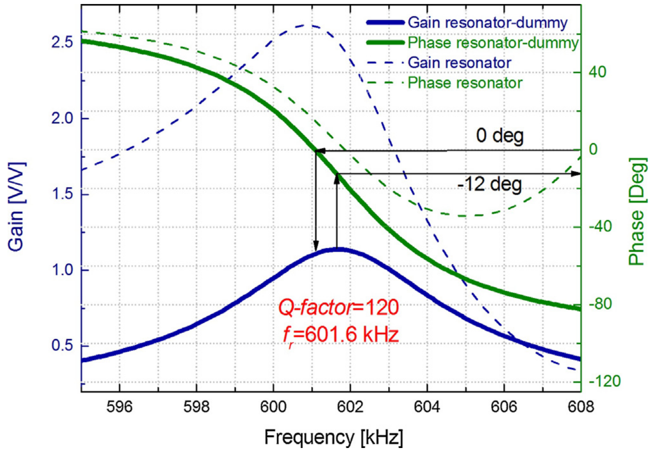

Fig. 2. Open loop response for the *15-mode* measured in a liquid test (2-Propanol) with and without the dummy compensation.

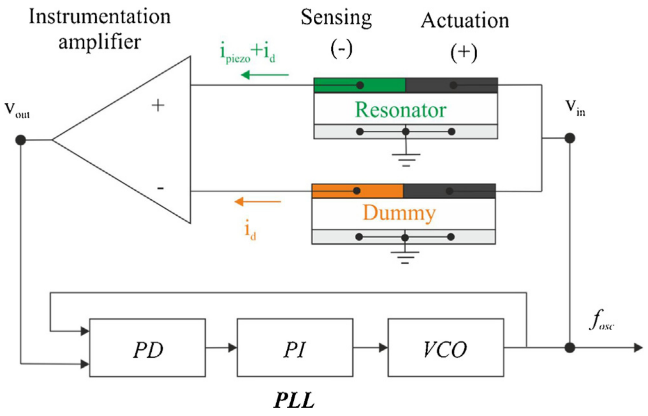

Fig. 3. Schematics of the PLL-based oscillator and the interface circuit.

In order to compensate this phase shift, the resonator and interface circuit were finally included in an oscillator based on a Phase Locked Loop (*PLL*) instrument (Fig. 3), instead of an oscillator circuit based on discrete components [16]. The phase compensation