TWO-STEP PROCEDURE FOR MULTI-MODE MEMS RESONATOR-BASED SENSING OF FLUID PROPERTIES

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ABSTRACT

In this paper, a novel approach for the determination of densities and viscosities of fluids, using a multi-mode MEMS resonator, is introduced. A self-actuating/self-sensing piezoelectric MEMS-resonator is design in operated in a distinct way, so that lateral as well as transversal bending modes can be excited within one measurement cycle. This enables a unique evaluation principle based on a two-step procedure. First, a special transversal bending mode with high *Q*-factors in liquids (e.g. $Q = 250$ in viscosity standard N1 from Paragon Scientific) is used to determine the density. As a second step, the lateral mode is excited to measure the viscosity, thus achieving deviations lower than 3% compared to the nominal values.

INTRODUCTION

Micro machined flexural resonators represent a vitally important class of analytical tools that have been used to provide physical information, such as the density and the viscosity of liquids [1-8]. Despite the high damping in such environments the sensitivity of these sensors is nowadays on a level, which qualifies them for a large variety of applications in e.g. the automotive [9] as well as in the food industry [10]. Nevertheless, in the determination of viscosity and density using transversally actuated, piezoelectric MEMS resonators, high deviations in the obtained viscosity values are reported [11]. On the other hand, pure lateral vibration modes lack in performance in high viscous liquids as the corresponding mechanical strain values are low, leading to a poor signal to noise ratio, when using piezoelectric read-out mechanism [3, 5, 12]. Furthermore, measuring pure shear forces by in-plane modes does not allow an independent determination of the density and viscosity [1]. However, the highly linearity of the quality factor over the inverse-viscosity-density-square-root product of lateral modes shows a promising basis of future evaluation methods. Therefore, a combination of different vibration modes is proposed to solve this issue, thus combining the beneficial properties of standard lateral and transversal modes.

EXPERIMENTAL DETAILS

Sensor characteristics

To investigate the impact of a two-mode evaluation procedure, a piezoelectric MEMS-resonator is fabricated such, so that lateral as well as transversal bending modes can be excited. In Figure 1, the mode shapes and the corresponding top views of the micro-machined sensor by finite-element method (FEM) Eigen mode analyses for the 6^{th}-order in (a) and for the first lateral mode in (b) are illustrated. Considering Leissa's nomenclature [13], by counting the number of nodal lines in x- and y-direction, the mode in Figure 1 (a) is named 17-mode. The corresponding antiparallel electrical actuation of the electrode stripes is indicated by the symbol "+" and "-", respectively. With such an actuation it is possible to actuate (a) transversal as well as (b) lateral bending modes. Furthermore, this tailored electrode design maximizes the collection of piezoelectrically generated charges of the same sign (+/−) without cancellation as it is described in detail in Refs. [7] and [14].

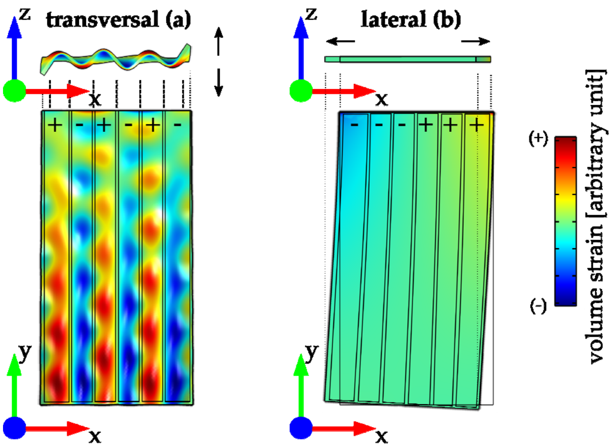

Figure 1: Visualization of the investigated transversal mode (17-mode) (a) and the lateral mode (b). The colored areas on the cantilever surface represent the local volume strain distribution.

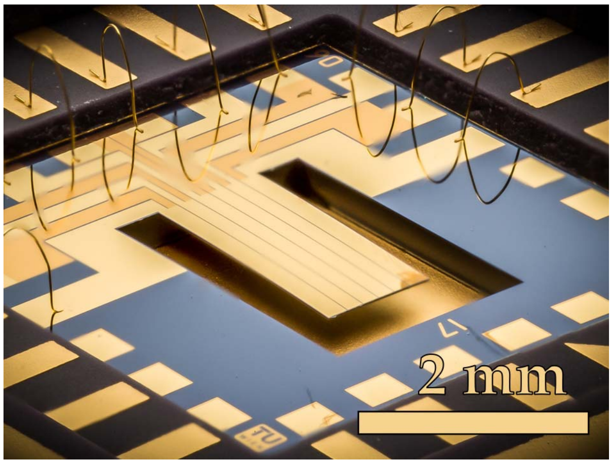

Figure 2: Optical micrograph of the in-house fabricated silicon die (6 x 6 mm²), containing one piezoelectric actuated plate (dimensions: 2524 x 1274 x 20 µm³) using an advanced electrode patterning considering the volume-strain of the modal shape presented in Figure 1.

In Figure 2 a typical die layout after mounting and wire-bonding with a released single-side clamped silicon plate (dimensions: length $L_{plate} = 2524$ µm; width $W_{plate} = 1274$ µm; thickness $T_{plate} = 20$ µm) is depicted. For actuating and sensing, an aluminum nitride (AlN) thin film with a thickness of $t_{AlN} = 1$ µm is sputter deposited being sandwiched between two separated $t_{be} = t_{te}$ = 50/450 nm thin chromium/gold thin films. A corresponding cross-sectional view of the sensor and its tailored electrode design, including detailed information about the sensor dimensions, are depicted in Figure 3. A detailed fabrication description is already published in Refs. [15] and [16].

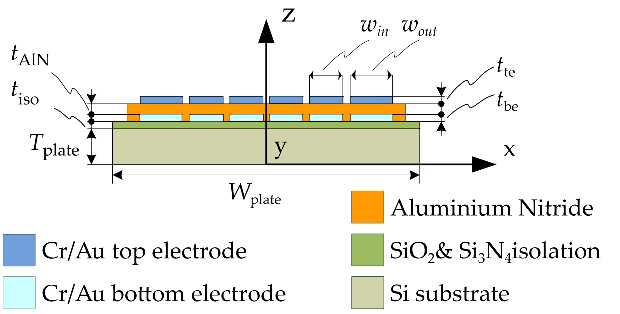

Figure 3: Schematic cross-sectional view on the MEMS resonator illustrating the electrode design and the plate support of the sensor with its dimensions: $W_{plate} = 1274$ µm, $T_{plate} = 20$ µm, $t_{iso} = 80/250$ nm, $t_{be} = t_{te} = 500$ nm, $t_{AlN} = 1$ µm and electrode widths of $w_{in} = 185$ µm and $w_{out} = 226$ µm, respectively.

Measurement procedure

Once the resonator is fabricated, a micropipette is used to cover the entire sensor surface with isopropanol and several viscosity standards from Paragon Scientific (N1, D5 and N10). To reduce the potential impact of air bubbles to a minimum, every measurement is performed twice. Due to the low electrical conductivity of the investigated fluids, a passivation of the sensor is not required. However, to guarantee comparable results, all further measurements were performed in a temperature-controlled environment at 20°C. The viscosity and density values of the investigated fluids are listed in Table 1. After each characterization step the MEMS resonator is cleaned with isopropanol and dried for 20 min at 20°C. Both, the lateral and transversal bending mode, are obtained electrically and optically using an Agilent 4294A impedance analyzer and a MSA-100-3D Laser Doppler vibrometer, respectively. A characteristic measurement of the lateral bending mode in isopropanol is depicted in Figure 4. Once the resonance frequencies of both, first lateral mode and 17-mode are localized, all further measurements can be obtained electrically (excitation voltage = 500 mV AC). Thereby, the actuation at resonance causes an increased average deflection and strained areas at the sensor surface, leading to an increased generation of polarization charges. As a consequence, an increased measurement current is detected by the impedance analyzer, and hence, an conductance peak ΔG results. This circumstance leads to the well-known characteristic resonance curves, whereas a typical representative is shown for the model solution N_1 in Figure 4 (b).

Density evaluation

As a first step the density ρ is obtained using the transversal 17-mode in combination with the well-known ansatz of a complex impedance, as described in Ref. [3] with D5 as calibration fluid. To do so, the resonator is first excited in air at +20°C and the corresponding resonance frequency and Q-factor are determined applying Butterworth–van Dyke equivalent circuit in combination with a Levenberg–Marquardt fitting algorithm [17]. The corresponding values for the equivalent circuit R_m, L_m and C_m, are given in Table 2 and are used to determine the densities of liquids as presented in Ref. [11].

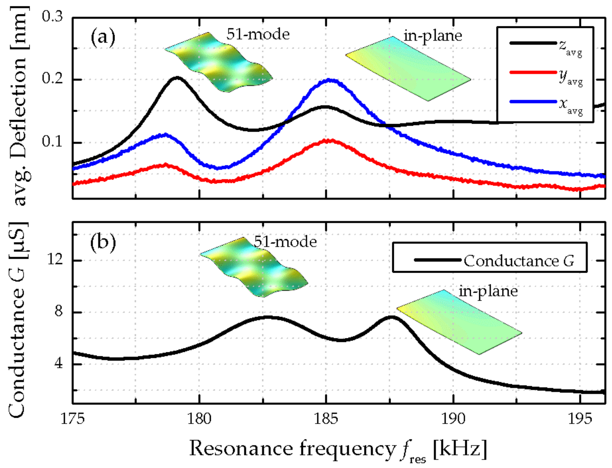

Figure 4: Optical Laser Doppler vibrometer measurements of the first in-plane mode in isopropanol at 20°C in (a) and the corresponding electrical impedance spectrum at 22°C in (b), obtained with a Polytec PSV-100-3D, and an Agilent 4294A impedance analyzer, respectively.

Table 1: Viscosity and density of Paragon Scientific viscosity standards N1, ISO, D5 and N10 at the measured temperature using the Ubbelohde Walther equation.

Sol.	Temp.	Density	Dyn. viscosity	1/•(•μ)
-	T [°C]	[g/ml]	μ [mPa·s]	
N1	20.5	0.777	1.029	1.119
ISO	20.6	0.781	2.454	0.722
D5	20.2	0.838	5.444	0.468
N10	20.5	0.850	17.453	0.260

Viscosity evaluation

When the sensor is excited laterally, the damping characteristic changes and the Q-factor changes linearly with $\sqrt{1/(\rho \cdot \mu)}$ for different liquids. This is used as a calibration curve for the unknown viscosities μ, as it is shown in Figure 5. To describe this dependency, a linear fitting function is used. Finally, the already known density values, obtained with the 17-mode are considered, so that the function Q_{fit} is now reduced to a function of the viscosity μ. Within this approach low deviations in the density as well as in the viscosity are obtained with respect to those given by the manufacturer of the viscosity and density standards, as listed in Table 3.

RESULTS

In Figure 1, finite element analyses are presented, showing the 17-mode in (a) and the first lateral bending mode in (b). From Figure 1 (a), it can be seen, that the nodal lines of the vibration fit to the pattering of the electrode stripes, so that a collection of all charges without cancellation is obtained. Nevertheless, by changing the actuation from (+-+-+-) to (---+++), as it is illustrated in Figure 1 (b), the first lateral bending mode can be actuated as well. In Figure 4 (a) the corresponding optical Laser Doppler vibrometer measurements are presented in a frequency range from 175 to 195 kHz.

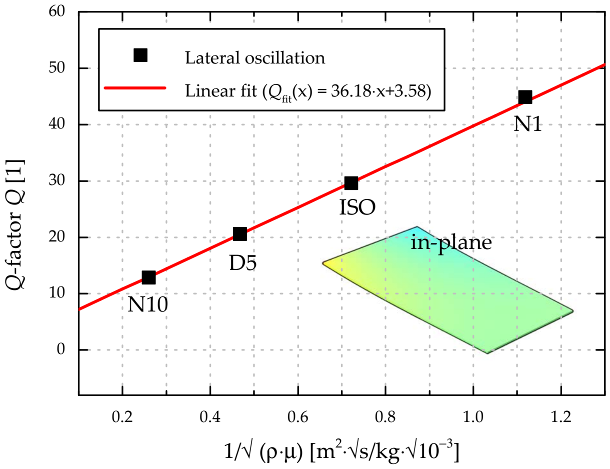

Figure 5: Q-factors over the inverse viscosity density square root product and a linear fit for several liquids (N1, Isopropanol, D5 and N10).

First, the optical measurement is used to localize the dedicated mode, as it can be seen for the first in-plane bending mode in Figure 4 (a), where the averaged lateral displacement X_{avi} exceeds the other two averaged displacements values in z- and y-direction. From this results it becomes clear that the first lateral bending mode is located around 185 kHz. For the sake of completeness, corresponding electrical measurements are presented below in Figure 4 (b). The resonance shift of ~ 5 kHz from optical to electrical measurement is due to different ambient temperatures of 20 and 22°C, respectively. These two measurements show, that it is possible to obtain valuable feedback from a piezoelectrically excited resonator in liquids, in a way that further evaluations can be performed. The second resonance peak in Figure 4 is the so called 51-mode and is not considered in this study.
The two-step evaluation procedure requires the characteristic values of the torsional exited, almost undamped resonator, which are given for an ambient temperature of 20°C in Table 2.

Table 2: Characteristic parameters of the resonator in air, including R_m, L_m, C_m.

f_{res} [kHz]	Q.[1]	R_m [k•]	L_m [mH]	C_m [fF]
2045	808.5	97.30	6.124	989.5

Using the evaluation procedure from Ref. [11] for the density, a minimal and maximal deviation of -0.71% (N10; ρ = 0.844 g/ml) and 4.03% (isopropanol; ρ = 0.0.812 g/ml) are obtained, respectively. As D5 is used for calibration, this deviation has to be 0%. In Figure 5 the results of the in-plane evaluation are presented, showing the expected linear dependency of the Q-factor over the inverse viscosity-density square root product for Q-factors between 10 and 45. Using linear fitting, a function $Q_{fit}(x) = 36.18x+3.58$ results. From this function $Q_{fit}(x)$, where $x = \sqrt{1/(\rho \cdot \mu)}$, and ρ is already gained by the previous evaluation, the unknown viscosity is calculated and listed in Table 3. In this case, the highest deviation of -3.16% was obtained for N1 with a dynamic viscosity of 0.996 mPa·s. These results show that the presented two-step evaluation procedure is suitable for the determination of densities and viscosities in a density range from 0.79 to 0.844 g/ml for the density and 0.996 to 17.998 mPa·s for the viscosity. Nevertheless, it has to be pointed out, that for higher viscous liquids the viscosity-density square root product will be significantly lower, which will lead to a lower responsively as μ is a function of $1/Q^2$.

Table 3: Measured densities and viscosities at 20°C and their corresponding deviations in percent when calibrated in D5.

Sol.	Density	dev.	Dyn. viscosity	dev.
-	[g/ml]	%	μ [mPa·s]	%
N1	0.791	1.81%	0.996	-3.16%
ISO	0.812	4.03%	2.436	-0.74%
D5	0.838	0.00%	5.491	0.86%
N10	0.844	-0.71%	17.998	3.13%

CONCLUSION

In this paper, a two-step procedure for the evaluation of densities and viscosities of liquids using a lateral and a transversal bending mode of a piezoelectrically excited MEMS-resonator is presented. First the transversal bending mode is used to determine the density of several liquids using an ansatz of a complex impedance. As a next step, the obtained densities are used in combination with results from the first lateral bending mode in a given liquid to determine the viscosities in a viscosity range from 1 up to 18 mPa·s. Thereby, low deviations in the density (lower than 4%) as well as in the viscosity values (lower than 3%) could be obtained. All in all, this study shows that the presented two-step procedure is suitable to determine the density and viscosity of low viscous liquids with low deviations when applying a one single-side clamped self-sensing/self-actuating MEMS resonator.

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