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J. Toledo · T. Manzaneque · J. Hernando-García ·
J. Vázquez · A. Ababneh · H. Seidel · M. Lapuerta ·
J. L. Sánchez-Rojas

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Abstract Real time monitoring of the physical properties of liquids is of great concern in the automotive industry. For example, tracking the viscosity of lubricating oils is of great importance because they are exposed to dilution with diesel fuel as result of late-injection processes, which are essential for regenerating diesel particulate filters. Here we describe two in-plane movement based resonators and their capability to assess oil dilution with diesel and biodiesel fuels. One of the resonators is a state-of-the-art micron-sized AlN-based rectangular plate, actuated in the first extensional mode in the MHz range. The second resonator is a commercially available millimeter-sized quartz tuning fork, working at 32.7 kHz. Electrical impedance measurements were performed to characterize the performance of both resonators in various liquid media over a wide range of viscosities. These measurements were compared with the results obtained with low-cost electronic circuits also developed in this work. In order to track density and viscosity of different fluids we have measured two parameters by various techniques: the resonance frequency and the quality factor.

1 Introduction

Monitoring the physical properties of liquids is of great interest in many fields. For example, in the automotive industry, the real-time monitoring of lubricating oils is of crucial importance to determine their properties. Lubricants are subjected to dilution with diesel fuel as a result of late-injection processes, which are necessary for regenerating diesel particulate filters (Lapuerta et al. 2012). This dilution can be determined by tracking the viscosity (μ) and density (ρ) of the dilution.

Resonant structures are an interesting option in the determination of the viscosity and density of liquids. Previous reports suggest the use of structures with out-of-plane movement (Tamika et al. 2007), although a more convenient approach is the use of structures with in-plane vibration modes (Jakoby et al. 2010). Here we consider the use of two in-plane movement resonators for the monitoring of oil dilution with diesel and biodiesel fuels (Fig. 1).

One of the resonators has been fabricated depositing AlN film as the piezoelectric actuator and it is a state-of-the-art micron-sized ($1,000 \mu\text{m} \times 250 \mu\text{m}$) rectangular plate, actuated in the first extensional mode in the MHz range (Manzaneque et al. 2012). The second resonator is a commercially available millimeter-sized quartz tuning fork (Momosaki 1997), working at 32.7 kHz. It is commonly used as a frequency standard in real time clock circuits. Tuning forks have also been reported as sensors for measurement of various physicochemical properties of liquids such as density and viscosity (Waszczuk et al. 2011).

With the goal of estimating the density and viscosity of a liquid, two key parameters characterizing the resonance have been measured: the quality factor and the resonant frequency. In a first stage, these parameters were derived from the electrical impedance spectra measured with a benchtop

J. Toledo (✉) · T. Manzaneque · J. Hernando-García ·
J. Vázquez · M. Lapuerta · J. L. Sánchez-Rojas
E.T.S.I. Industriales, Universidad de Castilla-La Mancha,
13071 Ciudad Real, Spain
e-mail: javier.toledo.serrano@gmail.com

A. Ababneh
Department of Electronics Engineering, Yarmouk University,
Irbid, Jordan

H. Seidel
Chair of Micromechanics, Microfluidics/Microactuators,
Saarland University, 66123 Saarbruecken, Germany

Fig. 1 **a** Millimeter-size tuning fork. **b** Micron-sized AlN-based rectangular plate. Picture on the *top* and measured modal displacement on the *bottom*

Fig. 2 Measurement setup with the impedance analyzer

analyzer. These preliminary results on the characterization of these two resonators, in various liquids, have been studied recently (Toledo et al. 2013). In this paper, specific electronic circuits have been implemented to obtain the same variables by means of a virtual instrument and a data acquisition card, resulting in a compact, low cost, real-time monitoring system. Although the resolution of these techniques may be enough in some cases, the impedance analyzer holds the lowest error in density and viscosity. With regard to the low-cost techniques, frequency response measurement and the transient analysis technique are compared in terms of the measurement error.

2 Measurement on fluids

2.1 Measurement setup

In order to register the performance of both resonators in different fluids a high precision Agilent 4294A impedance analyzer was employed. Besides typical solvents, viscosity standards provided by Paragon Scientific were used (Paragon). The measurement setup with the impedance analyzer is presented in Fig. 2.

In order to assure the full immersion of the resonators in the liquids under consideration, special cells were designed. In the case of the AlN resonator, the device is attached to a

Fig. 3 Liquid cell for **(a)** the tuning fork and **(b)** the AlN-based micro-resonator

printed circuit board (PCB), and microsoldering is used to connect the resonator electrodes to the copper pads of the PCB. Immersion is achieved by placing a drop of fluid on top of the device (Fig. 3). However, for the tuning fork we have used a simple test tube. The bottom of the test tube is removed and the tuning fork is inserted through the bottom aperture, while the two pins of the tuning fork remain outside for the electrical connection. Silicone is used to attach the base of the tuning fork to the test tube. The impedance measurements, for the fluids in Table 1, were developed at room temperature (25 °C) for both resonators (see Fig. 4).

In order to calculate the resonance frequency and the quality factor (see Table 1), the measured spectrum was fitted to a modified Butterworth-Van Dyke equivalent circuit

Table 1 Comparison of quality factor and resonance frequency for both resonators

Fluid	Tuning fork		AlN	
	Q	fr (Hz)	Q	fr (Hz)
Heptane	163.76	29,804.5	109.71	3,846,933.0
Methanol	133.87	29,400.0	86.90	3,837,045.6
Ethanol	86.18	29,299.5	71.43	3,830,251.6
2-Propanol	70.45	29,298.0	61.98	3,832,118.8
D5	46.92	29,033.9	53.66	3,819,374.6
N10	26.68	28,756.4	33.10	3,797,942.3
S20	17.87	28,460.0	24.95	3,782,421.3
N35	14.75	28,253.0	18.39	3,763,389.0

Fig. 4 Conductance measurements for the first anti-phase in-plane mode of the tuning fork (a) and for the first extensional mode of the AlN-based resonator (b). Vertical offsets were applied for clarity purposes. The Q -factor is also indicated in the legend

(Fig. 5) (Arnau et al. 2000). The RLC values are obtained by fitting the conductance of the circuit model to the experimental conductance spectrum around resonance. The

Fig. 5 Modified Butterworth-Van Dyke equivalent circuit. R_0 and C_0 represent the parasitic resistance and capacitance related to the piezoelectric layer and the R – L – C arm represents the motional behavior of the structure

parameters L and C are adjusted to minimize the difference between the calculated conductance and the experimental data for each frequency (Cassiede et al. 2010). Although there exists a variation of the parameters L and C , the value of the resonance frequency undergoes no or little deviation as it is a function of the product LC , and an increase in L reduces C and vice versa. The error obtained from various consecutive measurements in the case of the tuning fork resonator for L and C were 2.24 and 2.26 %, respectively. However, the error presented in the resonance frequency was 0.02 %.

$$\text{fr} = \frac{1}{2\pi\sqrt{LC}}, \quad Q = \frac{\omega L}{R} \quad (1)$$

2.2 Application of resonators as density–viscosity sensors

In order to assure the viability of both resonators as density–viscosity sensors, we have calculated both the quality factor and the square root of the product of the density and the viscosity of the liquids. A reasonable linear correlation is found (see Fig. 6), what demonstrates the viability of both resonators even for high viscous liquids. The fluids D5, N10, S20 and N35 correspond to viscosity standards by Paragon Scientific.

In order to estimate the density and viscosity values, a relationship between both quality factor and resonance frequency, against density and viscosity is required. Such study has been already reported for tuning fork resonators (Waszczuk et al. 2011; Zhang et al. 2002; Kokubun et al. 1984) and for extensional resonators (Castille et al. 2010; Dufour et al. 2014).

Following the procedure reported by Waszczuk et al. (2011) to solve the corresponding equations for both density and viscosity with the tuning fork, we have estimated two different constants, C_μ and $C_{\rho\mu}$, through a calibration from all the measurements previously carried out with the impedance analyzer, using various liquids whose viscosity and density are known (Wohlfarth and Wohlfahrt 2002).

Fig. 6 Quality factor of both resonators in various liquids

Table 2 Comparison between the estimated and the reported values of density and viscosity for different liquids as deduced by means of the impedance measurements obtained in the tuning fork

Fluid	Estimated values tuning fork (25 °C)		Reported values (25 °C)	
	μ_{est} (mPa s)	ρ_{est} (g/ml)	μ_{rep} (mPa s)	ρ_{rep} (g/ml)
Heptane	0.48	0.663	0.38	0.679
Methanol	0.65	0.767	0.54	0.792
Ethanol	1.29	0.780	1.04	0.785
2-Propanol	2.39	0.771	1.96	0.781
D5	5.19	0.818	4.74	0.835
N10	16.23	0.832	14.50	0.848
S20	37.07	0.836	36.92	0.855
N35	55.63	0.838	55.52	0.854

The values estimated for C_η and $C_{\rho\mu}$ were 1.02×10^{13} and 195.32, respectively, in the viscosity range of 0.5–56 cP. Table 2 shows the estimated values of density and viscosity for the different liquids used in Fig. 4. Theoretical values are also shown according to Wohlfarth Ch and Wohlfahrt (2002).

For the case of the extensional AIN resonator, assuming a hydrodynamic force determined by the Stokes's second problem approximation, both quality factor and resonance frequency depend on the density–viscosity product, preventing the resolution of these variables separately (Dufour et al. 2014). For the application of this resonator, the quality factor against the density–viscosity product has been fitted to a second order function in the reference liquids, determining coefficients in Eq. (2), with the reported density and viscosity values. This approximation can then be used to determine viscosity from the measured quality factor in other liquids, if we assume a known density, as shown in the next section.

$$aQ^2 + bQ + c = \frac{1}{\sqrt{\rho \cdot \mu}} \quad (2)$$

2.3 Density and viscosity measurements of lubricants and fuels

Our main goal is to apply these resonators to the analysis of the rheological properties of low-viscous automotive industrial fluids, such as lubricants and fuels.

In the case of the lubricants, we paid attention to their dilution with diesel fuel. An automotive lubricant oil such as SAE 15W40 was used and a commercial diesel fuel supplied by Cepsa was blended with the mentioned lubricant.

In the case of the fuels blends, it is of great importance to quantify the biodiesel content in biodiesel/diesel blends, since different contents will require a modification of diesel engine parameters for a clean and efficient combustion. To simulate different scenarios of biodiesel penetration, different diesel/biodiesel blends were prepared with biodiesel produced from animal fat, supplied by Stock del Valles S.L.

Table 3 shows the values of density and viscosity deduced from the calibration obtained in point 2.2 for different diesel/biodiesel blends. The same can be seen in Table 4 for the different lubricant/diesel mixtures prepared. Both tables indicate also the quality factor and resonance frequency for the two resonators under study. In the case of the extensional resonator, the density has been assumed to be constant to allow the determination of viscosity from the measured quality factor.

The measurements of the viscosity and density for lubricants and fuels, as specified in the ISO 3104, were developed at 40 °C. Densities were measured by means of hydrometers submerged in a thermostatic bath at 40 °C (Tamson TV2000) following the method established in the standard EN-ISO 3675. Viscosities were measured with modified Ostwald Cannon–Fenske type viscometers, using the same thermostatic bath at 40 °C, following standard EN-ISO 3104. As the viscosity increases, in both cases, there is a clear decrease of the quality factor that can be detected with the instrumentation.

3 Design of low-cost electronic circuits

All the previous measurements were taken with an impedance analyzer of high cost and dimensions. For applications requiring field or remote measurements, a compact, low-cost and precise enough approach is desirable (Niedermayer et al. 2009; Ferrari et al. 2010). Oscillators based on piezoelectric resonators may be a suitable choice, although their application with the low quality factors considered in this work is challenging, and only the frequency can be directly measured (Ferrari et al. 2008; Schröder et al. 2001).

Table 3 Density and viscosity values of blends of diesel and biodiesel fuels deduced by means of the measured frequency and quality factor

Blends (% vol)		Tuning fork		AIN		Tuning fork estimated values (40 °C)		AIN estimated values (40 °C)		Measured values (40 °C)	
Diesel	Biodiesel	Q	fr (Hz)	Q	fr (Hz)	μ_{est} (mPa s)	ρ_{est} (g/ml)	μ_{est} (mPa s)	ρ_{est} (g/ml)	μ_{mea} (mPa s)	ρ_{mea} (g/ml)
100	0	58.09	29,042.2	50.10	3,829,699	3.15	0.833	3.65	0.850	2.87	0.834
80	20	53.06	29,040.2	48.86	3,821,368	3.78	0.828	3.98	0.850	3.18	0.840
60	40	52.15	29,034.5	48.04	3,815,939	3.91	0.829	4.23	0.850	3.46	0.846
40	60	51.58	29,029.7	47.17	3,809,837	3.99	0.829	4.50	0.850	3.95	0.852
20	80	51.67	29,019.8	46.51	3,814,660	3.97	0.832	4.72	0.850	4.45	0.857
0	100	49.47	29,001.2	45.71	3,808,740	4.31	0.834	5.01	0.850	4.81	0.864

Measured values with laboratory instruments are also included

Table 4 Density and viscosity values of mixtures of lubricants and diesel fuel deduced by means of the measured frequency and quality factor

Mixtures (% vol)		Tuning fork		AIN		Tuning fork estimated values (40 °C)		AIN estimated values (40 °C)		Measured values (40 °C)	
Diesel	Lubricant	Q	fr (Hz)	Q	fr (Hz)	μ_{est} (mPa s)	ρ_{est} (g/ml)	μ_{est} (mPa s)	ρ_{est} (g/ml)	μ_{mea} (mPa s)	ρ_{mea} (g/ml)
0	100	10.65	27,834.0	12.71	371,4548	75.39	0.903	67.75	0.850	71.95	0.862
2	98	11.20	27,991.2	13.13	3,726,633	69.78	0.873	66.26	0.850	65.41	0.861
4	96	11.73	28,073.3	13.26	3,727,707	64.51	0.864	65.79	0.850	60.72	0.860
6	94	11.88	28,125.1	13.40	3,729,945	63.41	0.853	65.26	0.850	55.20	0.859
8	92	12.47	28,226.2	14.18	3,752,444	58.56	0.839	62.39	0.850	50.54	0.858
10	90	14.76	28,305.0	14.71	3,744,971	42.36	0.862	60.43	0.850	47.78	0.857

Measured values with laboratory instruments are also included

Fig. 7 Open-loop topology for the measurement of the resonance frequency and the quality factor

In order to obtain the density and the viscosity of the fluids under test, measurements of the resonance frequency and the quality factor were carried out with the development of a PC-based virtual instrument and low-cost electronic circuits. Once these two parameters are known, the viscosity of the fluids under test can be determined.

The design of the low-cost electronics is based on an open-loop topology (Fig. 7). This circuit features a transimpedance amplifier (Ferrari et al. 2001), necessary to convert the current signal delivered by the resonator to a voltage signal that is sampled by a data acquisition board (DAQ). The circuit was developed to measure the response of both the tuning fork and the AIN resonators actuated in

three different environments: air, 2-propanol and the viscosity standard by Paragon Scientific N35. The same measurements were complemented with an impedance analyzer in order to evaluate the performance of the open-loop electronic circuit.

3.1 Low-cost electronic circuits for the tuning fork resonator

The measurement of the quality factor and the resonant frequency can be performed applying two different techniques.

Fig. 8 Circuit diagram for the measurement of the resonance frequency and the quality factor with the tuning fork resonator

Fig. 9 Transient response in both air and 2-Propanol

Fig. 10 Frequency response function both in air and 2-Propanol

The first technique is based on a transient analysis (Rodahl et al. 1996). The excitation is performed with a multi-tone signal which was created with LABVIEW, from

National Instruments, and applied to the resonator through a DAQ card (Wali et al. 2010). The DAQ board was also used to read the voltage signal output of the transimpedance amplifier. The multiplexer A (MUX A) in Fig. 8 is controlled by a TTL signal generated by the DAQ board. This TTL signal switches the multiplexer every second triggering the DAQ samples during the exponential decay. In this case the DAQ board registers the output of the transimpedance amplifier. The response is finally fitted to an exponential decay in LabVIEW through the following equation.

$$A(t) = A_0 \cdot e^{-\frac{t}{\tau}} \cdot \sin(\omega t + \phi) \quad (3)$$

This fitting allowed us to obtain the decay time constant and the quality factor through the next equation. The resonance frequency has also been calculated with LabVIEW, dividing the number of peaks detected by the measured time interval (see Fig. 9).

$$Q = \frac{\tau \cdot \omega}{2} \quad (4)$$

The second technique is based on a frequency response function (FRF) (MUX A and MUX B closed in Fig. 8). After processing the data with LABVIEW, the values of the resonant frequency and the quality factor were obtained applying a method developed by Marshall and Brigham (2004). As it was reported, the Q -factor can be calculated from the impedance spectrum in the range of the mode peak according to:

$$Q = \frac{2f_r^2}{f_+^2 - f_-^2}, \text{ being } f_r = \sqrt{\frac{f_+^2 + f_-^2}{2}} \quad (5)$$

where f_- and f_+ are the frequencies that maximize and minimize the susceptance (i.e. imaginary part of admittance), respectively (Manzanares et al. 2010).

A remarkable difference in the value of the quality factor obtained in air using this technique and comparing it to

Table 5 Comparison of the different measurement techniques for both resonators

		Tuning fork			AIN		
		Air	2-Propanol	N35	Air	2-Propanol	N35
Frequency response	Q	976 ^a	75.5	19.5			
	ΔQ (%)	3.07	17.62	26.67			
	fr (Hz)	32,776	29,394	28,308	3,871,660	3,780,840	^b
	Δ fr (%)	0.02	0.23	0.69	0.89	0.02	
Transient response	Q	12,884	65.6	19.2	2,622	73.6	
	ΔQ (%)	4.59	8.23	34.90	4.50	9.24	
	fr (Hz)	32,767	29,494	28,542	3,864,400	3,805,760	
	Δ fr (%)	0.08	0.81	2.94	0.02	5.24	
Impedance analyzer	Q	13,150	70.4	14.7	2,397	61.9	18.3
	ΔQ (%)	6.39	3.41	2.04	9.60	10.34	19.13
	fr (Hz)	32,758	29,298	28,253	3,875,544	3,832,118	3,763,389
	Δ fr (%)	0.02	0.11	0.34	0.20	0.09	0.14

^a Discrepancy related to the fact that the approach of Marshall and Brigham (2004) handles only low figures of merit

^b Noise present in DAQ board

the impedance analyzer measurements can be observed in Table 5. The origin of this discrepancy may be related to the fact that the approach of Marshall et al. handles only low figures of merit, i.e. low Q values. For the cases of 2-propanol and N35, where the model of Marshall is valid, it has been demonstrated that the estimated quality factor agrees with the impedance analyzer measurement (Fig. 10).

3.2 Low-cost electronic circuits for the AIN resonator

The AIN structure resonates at a frequency (3.8 MHz) that cannot be generated with the DAQ card used in this work, whose maximum sampling rate is limited to 250 kS/s. For this reason a voltage-controlled oscillator (VCO) and a passive envelope detector were added to the system (Jakoby et al. 2005; Riesch and Jakoby 2007). The envelope detector includes a diode (BAT43), a resistance (1 M Ω) and a capacitance (100 pF). The VCO input is supplied with a sawtooth waveform generated with the DAQ card at a much lower frequency (Nakamoto and Kobayashi 1994). The sawtooth waveform frequency is related to the Q factor of the sensor. The biggest decay time constant for the AIN resonator (in heptane) is approximately 9 ms. It is clear that the period of the sawtooth waveform should be longer than the total decay time ($5\tau = 45$ ms). In order to avoid problems with the oscillation decay we have used a sawtooth frequency of 1 Hz.

$$\tau = \frac{2Q}{\omega} \quad (6)$$

In order to assure that the oscillator output frequency falls within the resonance frequency, different parameters of the VCO have been modified. A frequency counter (Agilent 53131A) was employed to determine the open-loop Allan deviation of the VCO, obtaining a value of 1.338×10^{-5} which is much lower than the width of the resonance peak for all the cases studied here, allowing to excite the resonator in a stable way (Rodriguez-Pardo et al. 2004).

The VCO utilized (74HCT4046) cannot deliver a sinusoidal waveform but a square wave signal instead. The

Fig. 12 a Frequency response in air, b Transient analysis in air

purpose of the low pass active filter shown in the schematic of Fig. 11 is to filter out all the harmonics present in the square wave. The resulting waveform is mostly sinusoidal and suitable to excite the microresonator. Finally, the envelope detector tracks the maximum output voltage of the transimpedance amplifier (Fan et al. 2011). As can be

Fig. 11 Circuit diagram for the AIN resonator

seen in Fig. 12, the response of the envelope detector peaks at $V_{in\_VCO} = 1.5$ V and helps to identify the resonance frequency value.

Once the desired VCO input voltage that allows an efficient excitation of the resonator was obtained, the same transient analysis technique that was utilized for the tuning fork resonator can be applied in this case. In this circuit the multiplexer is also controlled by a TTL signal. Besides, in order to obtain the resonant frequency more accurately, a logic comparator was added to the system. Its output is connected to the frequency counter included in the DAQ board. The relevant signals of the circuit are shown in Fig. 13.

In Table 5 the results obtained with the low-cost electronic circuits and the impedance analyzer in air, 2-propanol and N35 are compared. In order to obtain an accurate measurement of the resolution, each liquid was measured five times to yield the measurement error for each technique and each resonator.

The results demonstrate that the transient response and the frequency response techniques implemented in this work are an accurate method that can be used to determine both the quality factor and the resonant frequency in liquids. In the case of the AlN structure in N35 fluid it was hard to obtain an accurate measurement due to noise from the electronic circuit and the resolution limitation of the DAQ board.

Fig. 13 Signal timing: **a** signal control of the multiplexer, **b** transient impedance amplifier output, **c** logic comparator output, **d** envelope detector output

Finally, the density and viscosity resolutions were estimated only for the case of the tuning fork resonator (Waszczuk et al. 2011), since the models used for the AlN resonators could not separately solve for viscosity and density.

As can be seen in Table 6, it is clear that the impedance analyzer holds the lowest error in density and viscosity. However, with regard to the low-cost techniques, it can be observed that the frequency response technique outperforms the transient analysis technique in terms of the measurement error, being the error in the transient response approximately twice as much as in the frequency response for the viscosity value of the N35 fluid.

4 Conclusions

Quartz tuning forks and AlN-based extensional resonators have been characterized in liquids with a broad range of densities and viscosities. Two important parameters have been measured using an impedance analyzer: the quality factor and the resonant frequency. In order to measure the liquid viscosity and density, a calibration of a tuning fork sensor was performed using a theoretical model. For the AlN resonator, a simplified model was considered, allowing to obtain a relationship between quality factor and viscosity, when density is assumed to be constant.

Measurement results show a linear correlation over a very wide range of viscosities from 0.5 to 56 cP. In conclusion, we have demonstrated the performance of both resonators in oils and diesel mixtures as well as in diesel/biodiesel blends. The quality factor measured for both resonators was very similar for all fluids under test.

In this work, measurements were completed with the development of low-cost electronic circuits and a PC-based virtual instrument. Besides, the resonance frequency and the quality factor were deduced from both the transient response and the open loop frequency response. Once these two parameters are known, the viscosity and density of the fluids under test can be determined, requiring only a small amount of test liquid.

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Table 6 Resolution of the different measurement techniques for the tuning fork resonator

Fluid	Frequency response		Transient response		Impedance analyzer	
	ρ (g/ml)	η (mPa s)	ρ (g/ml)	μ (mPa s)	ρ (g/ml)	μ (mPa s)
2-Propanol	0.748 ± 0.025	2.08 ± 0.77	0.715 ± 0.065	2.71 ± 0.43	0.771 ± 0.008	2.39 ± 0.17
N35	0.894 ± 0.074	42 ± 7.5	0.888 ± 0.11	44.6 ± 14	0.838 ± 0.017	55.6 ± 3.2

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