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Accurate Determination of Viscosity and Mass Density of Fluids using a Piezoelectric Tuning Fork Resonant Sensor

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Abstract

Resonant sensors yield outstanding performance in the measurement of viscosity and mass density of fluids when signal conditioning, signal processing and thermal control are optimized and the fluid-mechanical model of the electromechanical resonator is suitable and accurate for the particular resonator. The combination of several advancements achieved in the last years significantly increases the performance achievable with this type of sensors. In this contribution the performance of a novel and highly universal evaluation system is demonstrated using a commercially available quartz crystal tuning fork resonator as sensing element for liquid viscosity and mass density. The obtained results are quantified with respect to an accurate lab bench viscosity and mass density meter. A significant advantage of this system is that it operates reliably and accurately even for very low quality factors (Q < 10). Therefore, the sensor elements can be used in a larger viscosity range than with alternative evaluation methods. Furthermore, the error propagation of the system can be estimated for each operating point, allowing the adaption of the measurement conditions (e.g. measurement time) to achieve a predefined accuracy. The implemented fully digital signal processing chain furthermore provides outstanding reproducibility of results, noise immunity and flexibility.

Key words: high accuracy, micro acoustic liquid sensor, low cross sensitivity, noise immunity

Introduction

Resonant sensors can be used in a wide range of applications, e.g. as microbalances, chemical sensors in liquid and gaseous environments, and for physical property sensing of liquid and viscoelastic media [1, 2]. Sensor elements with direct linear relation between the measured quantity and a processable output signal are desired in measurement practice, but are often not available, especially when high accuracy and suppression of cross influences are required. For a viscosity and mass density sensor, the utilization of a measurement principle which evaluates the frequency response of an electroacoustic resonator in contact with the fluid under test is advantageous. The frequency response of such a resonant sensor is related to the fluid properties by a nonlinear function but is also affected by several other – in most cases spurious – influences [3]. The most commonly known example for such an influence is the electrode capacitance of quartz crystal resonators (QCR). An established electrical model for this resonator type is the Butterworth - Van Dyke (BVD) circuit as shown in Fig. 1 below, comprising a series resonant RLC circuit, representing the electromechanical (motional) properties of the resonator, and a parallel capacitance between the electrodes C_0.

In the BVD model, an extension to a simple second order resonant system has to be considered in form of the parallel capacitance C_0. For alternative sensor concepts, e.g. using Lorentz-force excitation and inductive read-out [4, 5], similar extensions to a resonant circuit are required to model inductive crosstalk or wire resistances, for instance. It can be shown that these sensors can be described by circuits similar to the BVD model [6] and thus the approach described here is also applicable to a wider class of sensors. For the sake of convenience, this contribution focuses on

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piezoelectric resonators as an illustrative example.

Readout of resonant sensors

The use of oscillator circuits detuned by the measurement parameters is one of the most common approaches for the readout of resonant sensors and is particularly suitable for sensors with high Q–factors or when the damping of the resonance remains constant. This is the case for, e.g., quartz crystal microbalances (QCM) monitoring the deposed metal layer in vapor deposition lines. One of the major advantages of oscillators is that this approach allows for very cost-efficient implementations yielding a frequency as output parameter which, in principle, can be measured very accurately. A comprehensive review on oscillator circuits for resonant sensor applications is given by Arnau [7].

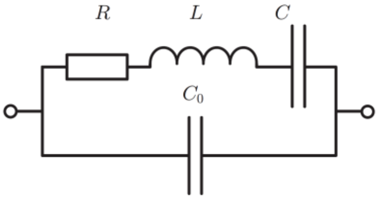

Fig. 1. Butterworth - Van Dyke model for QCR. The simple damped resonant circuit is paralleled by the electrode capacitance C_0 of the QCR .

For operating conditions where increased damping of the resonator is expected, e.g. when electromechanical resonators are exposed to liquid environments, additional effort has to be made to separate the behavior of the motional branch from parasitic effects like the parallel capacitance and the fluid’s permittivity and conductivity.

Several variations of locked loop circuits were reported to consider these influences e.g., [8-10]. In general, the locked loop circuits potentially yield a higher accuracy of the resulting measurements compared to oscillator circuits. This is due to the fact that the systems utilize more information on the resonator than simple oscillator circuits, the steering function implemented in the loop can suppress constant spurious influences. Spurious influences that are changing during operation can be addressed only to a very limited extent (e.g., compensation of changes in parallel capacitance as shown in [8]).

With increasing demands for measurement accuracy as well as with further increase of resonator damping, this will be more and more difficult to realize.

In order to obtain the best accuracy with significantly damped resonators, the only practicable solution is to record the resonators behavior in vicinity of the resonance and to shift the extraction of desired parameters to a post processing step in the digital domain. In the last decade, various approaches for dedicated analyzer systems were reported e.g., [11, 12], most of them designed to meet specific requirements in a specific application. By the use of measurement methods like those implemented in gain-phase / network / impedance analyzers, the information acquired from the resonator is significantly larger than with oscillator circuits or locked loop approaches.

For many applications the high complexity of resonant sensor systems is definitely an obstacle in designing high performance measurement systems. Like a chain being only as strong as its weakest link, each single link in the measurement system comprising resonator mechanics, frontend electronics, analog signal conditioning, data acquisition, signal processing and, last but not least, fluid mechanical models for the resonator-fluid interaction, has to be optimized individually but also must match the adjacent links to achieve optimal performance of the entire system.

In our previous work, we addressed numerous details in order to improve the performance of these compact analyzer systems. This concerns data acquisition concepts for minimal signal processing effort [12], approaches to reduce parasitic signal components [13], numerical methods for separating motional from parasitic behavior [14] as well as improvements in sensor modeling [15].

The latest development in this respect is a highly universal evaluation system for interfacing resonant sensors, which utilizes and combines various approaches that are required or simply beneficial for a high performance measurement system. This system is developed by the university spin-off Micro Resonant Technologies and tested in collaboration with the Institute for Microelectronics and Microsensors at the Johannes Kepler University Linz.

The first prototype of this new system is designed to operate at frequencies from DC to 100 kHz. It can be adapted to operate with virtual any type of resonator within the specified bandwidth, e.g. with resonators based on electromagnetic principles such as [4, 5] as well as with piezoelectric resonators like commercial off-the-shelf tuning fork or thickness shear mode resonators.

Tuning fork resonator as sensor for viscosity and density of fluids

Various publications address the use of tuning fork resonators for determination of physical fluid properties such as viscosity and mass density (e.g., [16-23]). In contrast to resonators with dominant shear oscillation (like torsional resonators or thickness shear mode QCR), the tuning fork resonator allows better separation of mass density and viscosity [6, 15, 23].

For the experiment presented below, we used a commercial off-the-shelf 32.768 kHz clock crystal where the housing was removed manually (Fig. 2). No electrical insulation of the sensor against the fluid is required, due to the implemented compensation of spurious effects, which efficiently eliminates cross-sensitivity to liquid permittivity and conductivity, spurious phase shifts and time delays.

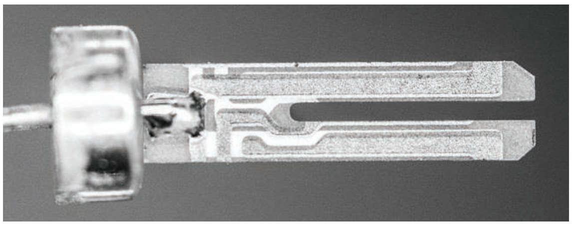

Fig. 2. Commercial off-the-shelf 32.768 kHz clock crystal with removed housing.

Experimental setup

These piezoelectric tuning fork resonators have very high motional impedance in the equivalent circuit [19] and therefore are prone to pick up noise by capacitive coupling. To minimize this impact, a low noise amplification stage was implemented close to the resonator (Fig. 3). The tuning fork is mounted on the cap of a small glass container and immersed in about 15 ml of the sample liquid. The container is inserted in a holder connected to a refrigerated and heated circulating bath system in order to accurately set the temperature of the sample to 25°C.

The amplifier is connected to the prototype of the new evaluation system through a standard twisted pair cable.

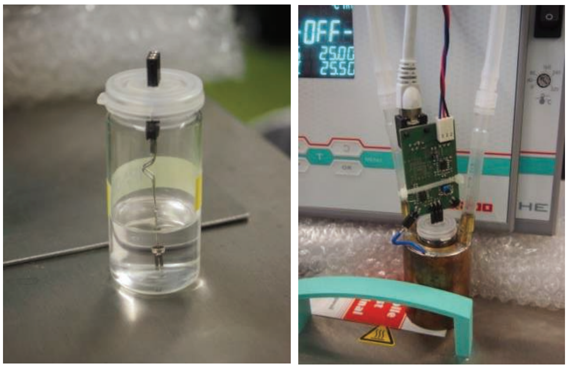

Fig. 3. Left: Tuning fork sensor in a glass container filled with approximately 15 ml of sample liquid. Right: Container with amplifier connected to the sensor terminals when inserted in the holder connected to the circulating bath via hoses.

The evaluation system is based on a digital signal processor (DSP) generating the excitation signal for the resonator and calculating the resonance frequency f_r and the quality factor Q from the recorded response signals by compensating for parasitic influences (like the parallel capacitance) and fitting the admittance of a series resonant circuit

$$Y = \frac{Y_{\max}}{1 + jQ\left(\frac{f}{f_r} - \frac{f_r}{f}\right)} \qquad (1)$$

to the remaining data, which is obtained after removing spurious signal components, by a multistage iterative optimization. From these parameters viscosity and density of the sample are determined by applying a fluid-mechanical sensor model of the tuning fork [15]. Finally the results are transferred to a host PC via USB port.

The measurement rate of the system is a parameter that can be set by the user within certain constraints, depending on the settling time (and hence the range of resonance frequency quality factor) of the resonator, and was set to approximately one measurement per second for this experiment. Increasing the measurement rate will increase the measurement noise on the obtained data.

Reference measurements for comparison purposes as well as for calibration of the resonator model were made with a Stabinger Viscometer (SVM3000) by Anton Paar, which nominally provides an accuracy of ±0.35% for viscosity and ±0.5 kg/m³ for mass density.

Results

Various test liquids with viscosities in the range of about 0.5-8 mPa s and densities in the range of about 780-930 kg/m³ were measured with the reference instrument and the novel resonant sensor setup.

Prior to each measurement run the sensor was rinsed with cleaner's naphtha and 2-propanol and dried with air. The resonator was inserted in the sample container placed in the holder sufficiently long to stabilize the temperature of the sample. Finally a sequence of 300 measurement points was acquired in about 5 minutes (exemplarily shown in Fig. 4).

From the acquired sequences mean values and standard deviations were derived and compared to the reference measurements. The distributions of the measurement results resemble Gaussian distributions (e.g., shown by chi²-test and kurtosis analysis). The variances of frequency and Q-factor and subsequently of viscosity and density results are in close agreement with the theoretical predictions for the error propagation of equivalent input noise through the whole signal chain. It could further be shown that our approach yields results virtually identical to the Cramer-Rao lower bound [24], when the distribution of the processed spectral lines is optimized iteratively.

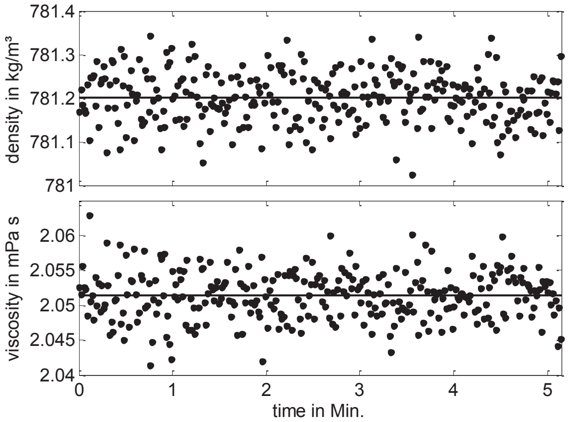

Fig. 4 Measurement in 2-Propanol after stabiliza-tion of temperature. With a measurement rate of about one measurement per second, 300 data points were acquired in a time of approximately 5 minutes.

A comparison of reference data and the mean values of the measurement results obtained with the tuning fork are shown in Tab. 1. As depicted in Fig. 5, these results deviate from the reference measurements less than 0.5 % for viscosity and less than 0.11 % for density.

Conclusions

A sophisticated viscosity and density measurement system is presented. For demonstration purposes, the system comprises a commercial off-the-shelf resonator as sensor, a frontend amplifier stage and a novel evaluation system for resonant sensors.

The results obtained with this setup are compared to an accurate top-grade lab bench viscosity and density meter and show outstanding trueness and precision at a significantly higher measurement speed, ideally suited for a large range of applications, such as online process monitoring, low fluid volume measurements, hand-held devices, laboratory use, etc.

Taking into account that the presented experiment was conducted with the first prototype of the new system we are confident to reach a performance comparable to top level lab viscometers.

Tab. 1: Measured values for viscosity and mass density compared to reference data (obtained with an Anton Paar SVM3000).

Sample	reference (SVM3000)		tuning fork sensor	
	viscosity mPa s	density kg/m³	viscosity mPa s	density kg/m³
Ethanol	1.0330	784.8	1.0317	784.76
2-Propanol	2.0531	780.4	2.0514	781.20
1-Pentanol	3.4816	810.1	3.4689	810.55
1-Octanol	7.6530	821.2	7.6526	821.20
RT5 oil	5.0336	911.8	5.0372	911.77
TEOS (Tetraethyl orthosilicate)	0.6022	927.7	0.6010	928.47
Toluene	0.4821	861.4	0.4842	860.48
Diesel	3.3740	831.6	3.3822	831.06
Diesel + 5% Toluene	2.8200	832.8	2.8229	832.32

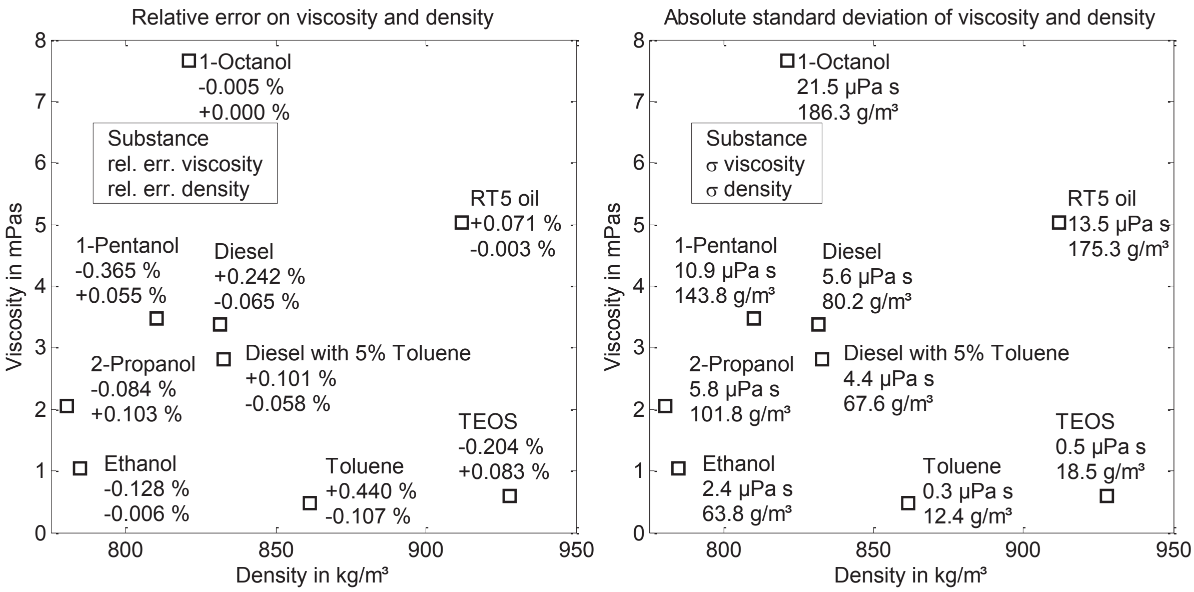

Fig. 5 Tested sample liquids with different density and viscosity. Relative errors and standard deviations are given for each sample.

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