A Feasibility Study on Tunable Resonators for Rheological Measurements

M. Heinisch¹, E.K. Reichel² and B. Jakoby¹

¹Institute for Microelectronics and Microsensors, Johannes Kepler University, 4040 Linz, Austria
²Center for Surface Science and Catalysis, Katholieke Universiteit Leuven, 3001 Heverlee, Belgium

A feasibility study on resonating sensors for rheologic properties such as viscosity, facilitating measurements at tunable frequencies in the low kHz range is presented. Results from analytical models relating measurement data to the viscosity are compared to measurement results from sample liquids. The benefits of the presented sensor designs in terms of high tunability and their sensitivity to viscosity are briefly discussed.

Introduction

The emphasis of the work summarized here is on miniaturized, tunable, resonating rheometers in which we presently focus on two main aspects: Large penetration depths (of shear waves which are excited in the examined liquid by the resonator) and high tunability (of the resonator’s resonance frequency). To allow for large penetration depths (several micrometers in case of aqueous liquids), the oscillator’s resonance frequencies should be in the low kilohertz range. Large penetration depths become important when examining complex liquids such as multi-phase fluids such as, e.g., emulsions. For the investigation of liquids showing shear thinning (or thickening) or viscoelastic behavior, it is necessary to record the liquid’s characteristics not only at one single frequency but in a range of different frequencies, which in the best case should cover several decades of resonance frequencies. For this purpose, especially in the case of resonating micro-sensors, it is desirable to have devices which can be operated at tunable frequencies without changing their geometries.

For this purpose, we designed and investigated two different types of tunable resonating sensors: A wire viscometer and a suspended plate rheometer (Fig. 1). For both concepts reviewed in this work, the ability of tuning the sensor’s resonance frequency is based on varying the normal stresses within (resonating) tungsten wires. In both cases, sample liquids are subjected to time harmonic shear stresses induced by the resonating wire and the suspended resonating platelet, respectively. From the resulting frequency response the liquid’s rheological properties can be deduced by fitting the parameters of an appropriate closed-form model representing the physical behavior of the sensors.

The use of appropriate materials and different micro-fabrication techniques were investigated and the applicability of the devices for rheological measurements was examined. In this contribution, the results from analytical closed form models, which were presented in [1] and [2], are compared to measurement results, and the capability of the particular resonator for accurate and reliable sensing is discussed.


**Experimental Setups**

Figure 1 shows the schematic drawings of the investigated sensor principles. The figure on the left shows the basic design of the wire viscometer. A 100 μm thick tungsten wire carrying sinusoidal currents is placed in an external magnetic field (not depicted) and thus oscillating due to Lorentz forces. For excitation, a function generator is connected to two 24 Ω resistors being connected in series with the tungsten wire. These resistors are used to limit the excitation current. For measuring the motion-induced voltage as read-out, a lock-in amplifier is used. On the right the suspended plate rheometer is illustrated. The sensor consists of two parallel wires placed in an external magnetic field $B$. In this figure, the left wire is used for exciting lateral vibrations by means of Lorentz forces. The second wire on the right used for pick-up is coupled to the excitation wire with a rigid plate, thus following the movement of the left wire. The induced voltage in the second wire is used as read-out. The platelet inducing (one-dimensional) shear waves into the liquid is the main part interacting with the sample. For both types of sensors, the resonance frequency can be changed by (mechanically) varying the normal stresses in the wires.

**Measurements**

**Measurement Principle**

For both types of sensors, their frequency response is recorded by sweeping the sinusoidal excitation current’s frequency in the bandwidth of interest (containing the fundamental harmonic) and recording the (motion-) induced voltage with a lock-in amplifier. Once all the parameters of the analytical closed form model (except the fluid’s dynamic viscosity and mass density) were identified with a set of calibration measurements, the liquid’s viscosity can be determined by fitting the model into the measured resonance curve (already knowing the liquid’s mass density which was e.g., evaluated by another measurement).

**Electrical Cross Talk**

In first measurements electrical cross talk was observed (Fig. 2 (left)). In air this electrical cross talk does not have a significant effect on the signal close to the fundamental harmonic. However, when measuring liquids, this effect can become disturbing. The
wires of the sensor connected to the wires for power supply and measuring form two loops (single-turn coils) with a certain cross-sectional area (Fig. 2 (right)). The magnetic flux generated by the current in the first loop penetrating the second loop induces a voltage in this second loop (Faraday’s law) and vice versa. Displacing the wires mostly changes the cross-sectional areas of these loops and thus, resulting in a different induced voltage resulting from this electrical cross talk, making reliable measurements difficult. To reduce the effect of electrical cross talk, BNC-connectors are used, first, to minimize the effective cross-sectional area of the single-turn loops, and second, to keep this cross-sectional area constant, enabling reliable measurement with strongly reduced and constant electrical cross talk.

![Fig. 2: Effect and explanation of electrical cross talk.](image)

**Tunability**

The band width of achievable resonance frequencies (in air) using 3 cm long and 100 μm thick tungsten wires were investigated (Fig. 3). In each case a detailed view of one characteristic resonance is depicted as well. In case off the wire viscometer (left) higher quality factors were obtained. (Note that the depicted bandwidths in the detailed views are 40 Hz for the wire viscometer and 200 Hz for the suspended plate rheometer.) The obtained resonances in these first measurements go from 1000 Hz to 4250 Hz in case of the wire viscometer and from 820 Hz to 4040 Hz for the suspended plate rheometer. In both cases, for changing the resonance frequencies for these measurements, the normal stresses in the wires were varied by tensioning the wires with micro-stages with micrometer screws.

To further investigate the tunable range of a wire when changing both, the normal stresses in the wire as well as its length, a setup as depicted in Fig. 4 was used. The conductive blades allow for electrical contact and for changing the wire’s vibrating length. One end of the wire is rigidly affixed. The other is attached to a weight, which is varied for changing the normal stresses in the wire but keeping the normal stresses constant when changing the vibrating length only. On the right hand side of Fig. 4 the results from a theoretic estimation of achievable resonance frequencies, when changing the normal stresses as well as the wire’s vibrating length for a 100 μm thick tungsten wire with lengths between 10 mm and 30 mm and normal stresses between 0 and 1.4 GPa, is depicted. (Note: Tungsten features yield stresses of 1.5 GPa, approximately.) These theoretical values were compared with first measuring results, and good accordance was observed.
**Response to Viscosity**

The response to viscosity and the sensitivity to the latter were investigated by immersing the sensors into different sample liquids. Figure 5 shows a comparison of theoretic and measured data for four different liquids. (Acetone: mass-density $\rho = 0.78 \text{ g/cm}^3$, dynamic viscosity $\mu = 0.21 \text{ mPas}$; Methanol: $\rho = 0.79 \text{ g/cm}^3$, $\mu = 0.47 \text{ mPas}$; DI-water: $\rho = 1 \text{ g/cm}^3$, $\mu = 0.86 \text{ mPas}$; Isopropanol: $\rho = 0.78 \text{ g/cm}^3$, $\mu = 2.05 \text{ mPas}$; values measured at 25 °C with an Anton Paar ® SVM3000).

In this case the relative diameter of the resonance circle in the Nyquist plot in respect to DI-water was used as quantity, which relates to the dynamic viscosity. The solid lines represent the theoretic values gained from the models in respect of the according mass density. The dots are results from measurements. A deviation of the predicted (theoretical) values from the measured values is observed, which might be due to drawbacks in the setup and / or the modeling. However, the calculated curve and the measured points show the same behavior.
Fig. 5: Comparison between measured and theoretic data.

**Conclusion and Outlook**

Both of the presented principles seem to be well suited for measuring a liquid’s viscosity in a large tunable range of resonance frequencies. Both principles show approximately the same sensitivity to viscosity as well as similar ranges of tunability. Thus, at this stage, it is hardly possible to meaningfully judge the benefits of one design over the other.

Regarding future work, we will comprehensively investigate the tunable range when varying both the normal stresses and the effective length of the vibrating mechanism. Furthermore, we will work on improving our setups for more reliable and accurate measurements as well on refining our analytical models to facilitate more accurate sensing.

**Acknowledgements**

We are indebted to the Austrian Competence Centre of Mechatronics (ACCM) for the financial support. We also want to thank Prof. Isabelle Dufour from Université de Bordeaux, Laboratoire de l’Intégration du Matériau au Système, France, for the very interesting and enriching discussions.

**References**
