Fluidic Sensors Based on Vibrating Miniaturized Devices

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***Abstract*—This paper reviews our recent work on vibrating sensors for the physical properties of fluids, particularly viscosity and density. Several device designs and the associated properties, specifically with respect to the sensed rheological domain are discussed.**

I. Introduction

Miniaturized sensors for physical liquid parameters can be utilized in applications where liquids in industrial processes have to be monitored in order to maintain the quality of a process or the associated product. Very often chemical sensors are applied for that purpose. Conventional chemical sensors feature a chemical interface layer attached to a physical transducer. The interface selectively reacts with the targeted chemical species changing its physical properties (e.g., mass density), which is transformed into an electrical signal by the attached transducer. Due to the adverse properties commonly associated with chemical interfaces (lacking reversibility, drift, etc.), sensing physical parameters as indicators for the state of the liquid represents an attractive alternative to conventional chemical sensors. As in the case of physical sensors the selectivity associated with the chemical interface is lost, either an array of physical sensors can to be applied (which, allows to create a state space that can be related to the targeted chemical property) or ab initio knowledge about the process being monitored can be used. An example for the latter would be the determination of the alcohol content in a brewing process by means of a liquid density measurement. This concept can be referred to as the implementation of "physical chemosensors" [1]. For liquid monitoring, suitable physical parameters are in particular the density ρ and the viscosity η of the liquid. Before we move on to discuss specific sensors, some simple facts on viscosity and rheology, i.e. the science of flow in matter, will be reviewed in the following (see, e.g., [2] for a detailed introduction into rheology).

The viscosity (more precisely the "shear viscosity") η can be defined in terms of a simple experimental arrangement, where the liquid under test is sheared between two laterally moving plates. η is then defined as the ratio between the applied shear stress (maintaining the movement) and the resulting gradient of the flow velocity (i.e. the so-called shear rate) of the liquid sheared between the plates. The so defined viscosity (as already proposed by Newton) can be measured in a straightforward manner by laboratory instruments, which in some manner impress a shear deformation on the liquid and measure the associated externally applied shear forces. Most often, rotational movements are utilized, e.g., by immersing a rotating cylinder in a viscous liquid. The ratio of applied torque and the resulting angular rate is then a measure for η. As an alternative to continuous rotation, also rotational harmonic oscillations can be applied. For so-called non-Newtonian liquids, the ratio of torque and angular rate (or equivalently, shear stress to velocity gradient) depends on the measurement parameters used. For example, the viscosity may drop for increasing shear rates (so called shear-thinning behavior). In case of rotational oscillations, the obtained viscosity may depend on the frequency and furthermore frequency-depending phase shifts between torque and angular rate can occur, which can be represented as an imaginary part in the viscosity when using complex notation for time-harmonic signals. The latter effect represents so called visco-elastic behavior. These two examples represent non-linear and linear distortions of the ideal Newtonian behavior.

The miniaturization of suitable viscosity measurement principles on the one hand facilitates the implementation of these devices online. On the other hand, scaling effect have to be taken into account, which lead to issues when it comes to applications in complex liquids such as suspensions featuring non-Newtonian behavior, which, as mentioned above, yields a dependence of the measured viscosity on the measurement parameters. This has led us to the investigation of different device designs, which are discussed further below in this contribution. Examples for monitoring processes utilizing viscosity sensors in our recent work would be the monitoring of transitions in emulsions [3], the monitoring of the deterioration of engine oil [4], and the monitoring of zeolite synthesis [5].

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II. THICKNESS SHEAR MODE RESONATORS

A. Device Principle

A very established way to determine the viscosity with a miniaturized sensor is the utilization of a thickness shear mode resonator. These devices basically consist of quartz disks, which are provided with electrodes on both faces of the disk. Applying an AC voltage to the electrodes, by means of the piezoelectric effect, excites vibrations in the disk. Depending on the crystal cut, different vibrational modes can be excited at their respective resonance frequency. For viscosity sensing, the thickness shear mode is particularly interesting, which is facilitated, e.g., by using the so-called AT-cut of quartz [6]. If the device is immersed in a liquid, the shear vibrations at the device surface entrain the liquid with the shear movement. For an ideal, non-viscous liquid, no such entrainment would take place as a non-viscous liquid cannot support shear stresses. In case of viscous liquids, however, a strongly attenuated shear wave is excited into the liquid. The decay length δ of this wave depends on the viscosity η, the density ρ, and the frequency ω:

$$\delta = \sqrt{\frac{2\eta}{\omega\rho}}\,. \tag{1}$$

The interaction with the liquid is also represented in the equivalent circuit related to the impedance between the electrodes. Due to piezoelectricity, close to resonance the mechanical resonance can be represented as a resonant LC circuit, e.g., as a so-called motional arm representing an LC series resonant circuit in the Butterworth-Van Dyke equivalent circuit [7]. The liquid loading leads to an additional inductance and resistor in this motional arm representing the entrained additional mass and the viscous damping of the device, respectively. For loading with a Newtonian liquid, both of these parameters are to first order proportional to $\sqrt{\eta\rho}$ [6].

B. Modeling TSM resonators

There are closed form solutions for TSM-resonators immersed in liquids [6], which, however, are based on a 1D-model and thus are not capable of accounting for spurious effects such as the unwanted excitation of compressional waves. The latter propagate with comparatively low attenauation even in viscous liquids leading to potential unwanted interference effects if they are reflected by nearby obstacles. The compressional waves are excited by vibrational normal displacements at the sensor surface which are due to a number of causes such as the non-uniform shear distribution in combination with restraints due to the conservation of mass (in the liquid as well as in the quartz) and the uncompensated angular momentum of the quartz disk [7],[9],[10].

In order to analyze these effects, the application of numerical methods such as the finite element method (FEM) seems near at hand. However, the characteristic length scales involved in the problem cover a fairly wide range: the attenuated shear waves show decay length which may lie in the submicron range, whereas the characteristic decay length of compressional waves can be in the order of 10 cm or more. This leads to a huge number of required discretization elements and thus to large computational costs. Alternatively the spectral domain method can be employed. This method, which assumes time-harmonic fields, is well-established in computational electromagnetics and also in the analysis of surface wave propagation. We will briefly outline the approach in the following in a very condensed fashion, a more detailed introduction is provided in a recent review paper [11].

Basically the method is applicable if layered structures are present. By neglecting end effects at the quartz disk, we may replace the disk by a laterally infinitely extended quartz slab (the method is thus only applicable to plano-plano quartz resonators). First, partial differential equations for the electromechanical fields (i.e., electric fields and mechanical stress and displacements) within the quartz and the adjacent liquid are established (the electrodes residing in the quartz-liquid interfaces are assumed to be infinitely thin and are not considered for the moment), where the Navier-Stokes equations governing the mechanical fields in the liquid are linearized. This is permissible if vibrational movements with small amplitudes are considered [10]. Next, a spatial Fourier transform with respect to the lateral coordinates (say x and y) is applied to obtain a spectral domain representation, where – due to the lateral uniformity of the involved materials (quartz and liquids) – differential operators with respect to x and y are represented by simple algebraic multiplications with the associated spectral variables k_x and k_y (these carry the dimensions of wavenumbers). The resulting equations can be cast into a linear first order system of differential equations with respect to a single spatial variable corresponding to the surface normal coordinate, i.e. z. This system can be solved by the usual exponential ansatz, yielding a representation of the fields in terms of eigenmodes within each uniform domain (layer) of the problem (in our case the quartz slab and the adjacent half spaces filled with liquids). Note that the electrodes in the interface between quartz and liquid have thus far not been considered as they represent a non-uniformity in the lateral directions x and y. Hence the approach thus far only yields a spectral domain representation for the fields in the quartz and liquid.

If an AC-voltage is applied to the electrodes, a charge distribution on the electrodes will be established, which, in turn, gives rise to the electromechanical fields that we want to obtain. To solve for the charge distribution, a numerical Method-of-Moments (MoM) approach is applied [12]. Here, the unknown charge distribution is approximated by a finite set of so-called basis functions (e.g., a set of rectangular functions next to each other covering the electrode region), which are each weighted (multiplied) by a so far unknown coefficient χ_i. Due to the assumed lateral uniformity of the surrounding media, the fields due to one of the basis functions can be obtained by a spatial convolution integral involving the basis function and the solution for the fields due to a point charge in the interface where the electrode resides. The fields due to a point charge are the so-called Green's functions and the can be easily determined in spectral domain as a Dirac delta function (representing a point charge) simply turns into a constant upon applying a Fourier transform. The spectral domain Green's functions are obtained by using the spectral expansions for the field discussed above and imposing the interface conditions at the quartz-liquid-interface (i.e. continuous fields except for a jump in the electric displacement due to the charge at the interface) and radiation conditions at infinity (i.e., considering only outward traveling eigenmodes in $\pm z$ directions). Superposing the fields for all the basis functions (with the associated coefficients χ as discrete variables), the boundary conditions (basically imposing constant potentials on the electrodes, the mechanical interface conditions have already been incorporated in the Green's functions) can be approximately established in weighted manner by using testing functions. In that manner, a linear system of equations for the coefficients χ is obtained. Once a solution for the χ has been obtained, the associated fields can be calculated in a post-processing step (again using convolution integrals and appropriate Green's functions).

This method requires some efforts in determining the eigenmodes, establishing the required Green's functions, and solving the numerical MoM-system, but once it is implemented it is numerically very efficient. In [13] some illustrative results with a particular focus on the spurious compressional wave excitation are presented.

C. Sensing Complex Liquids with TSM resonators

For sensing viscosity with TSM resonators, the utilized principle is basically comparable to oscillatory rheometers. However, the used parameter range (characterizing the probed "rheological domain") is very different: TSM resonators commonly oscillate in the MHz regime whereas oscillatory rheometers work in the range of a few Hz and below. Furthermore, TSM resonators feature displacement amplitudes in the nanometer-range compared to millimeters for the laboratory viscometers. The employed high frequencies in the case of TSM resonators also cause a small penetration of the involved shear wave (typically with δ being in the micron to submicron range) such that basically only a thin film of liquid is being probed. One may thus speak of an "RF-thin-film-viscosity" that is being determined. This has a severe impact on the obtained results when complex liquids are being sensed. A simple example would be emulsions, i.e. water droplets dispersed in oil (W/O-emulsions) or oil droplets in water (O/W-emulsions). In recent works we have investigated that for common emulsions (which will be referred to as macroemulsions in the following) featuring droplet sizes of 10 microns or more, which are thus larger than the penetration depth δ of the shear wave. The impact of the presence of the droplets on the (macroscopically) apparent viscosity of the emulsion is thus not detected by the sensor; it rather measures the viscosity of the continuous phase, in general. In the case that a droplet adheres to the surface and thus covers a fraction of it, some kind of average viscosity of oil and water rather than the macrocsopically apparent viscosity of the emulsion is obtained: in the region where the droplet adheres, the viscosity of the medium representing the dispersed phase is being sensed, while elsewhere on the surface the viscosity of the continuous phase determines the interaction with the vibrating sensor surface. Confirming these considerations, this behavior actually disappears as soon as the average droplet size becomes smaller than the penetration depth δ which was confirmed in experiments with so-called microemulsions, where – thanks to special surfactants – droplet sizes in the submicron-range are achieved [14], see also Fig. 1.

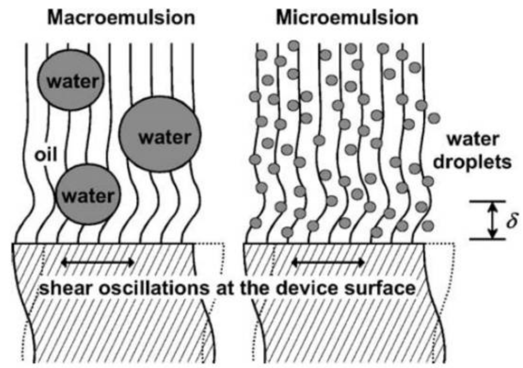


Fig. 1. Sensing macro- and microemulsions with TSM-resonators.

Using microemulsions, another impact of the limited penetration depth can be demonstrated. While for macro-emulsions, depending on the mixing ration of water and oil, the microstructure always represents either water droplets dispersed in a continuous oil phase or vice versa, for microemulsions so-called bicontinuous phases can occur for intermediate mixing ratios. There, both phases are represented by continuous phases which are entangled in a sponge-like manner [3]. Upon appearance of these bicontinuous phases, the macroscopically apparent viscosity increases. As the onset of a bicontinuous phase represents a significant extension of the characteristic dimensions associated with the microstructure, the TSM sensor again does not sense the macroscopically apparent viscosity. This is illustrated in Fig. 2 comparing the viscosities measured with a laboratory viscometer to those determined with a TSM-resonator for a continuous increase of the aqueous phase of a specifically considered model-microemulsion, where bicontinuous phases appear for intermediate aqueous phase content as indicated on the x-axis [3].

Thus, in summary, one has to be cautious when sensing complex, i.e. non-Newtonian liquids with TSM-resonators, as the obtained viscosity ("RF-thin-film-viscosity") will in general not represent that obtained by lab viscometers ("macroscopically apparent viscosity"). The devices described further below have been designed by particularly aiming at probing a rheological domain that is closer to that of lab viscometers where the miniaturized character (needed for implementation in online monitoring systems) is still maintained.

D. Electronic Readout of TSM resonators

When applying TSM sensors in viscous liquids, depending on the viscosity, low Q-values may prevail. Thus the common readout method of using the resonator as frequency-determining element in an oscillator, which is near at hand and is frequently used, e.g., in chemical gas sensing applications, may not be applicable as the oscillation may become noise or may not be sustainable at all. Thus alternative approaches are based on implementing miniaturized impedance analyzers ranging from purely analog to largely digitized concepts as described, e.g., in [15],[16],[17],[18].

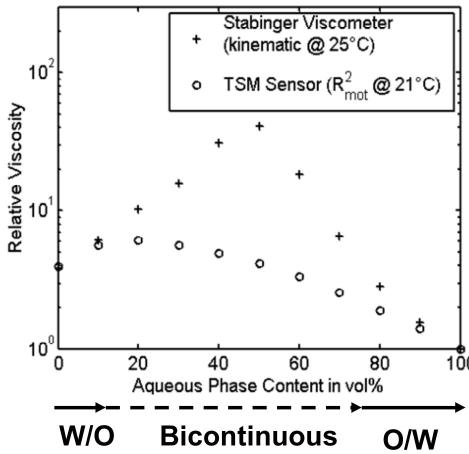


Fig. **2**. The appearance of bicontinuous states in microemulsions and its effect on the viscosity determined by means of TSM-resonators compared to the viscosity of a lab viscometer.

III. Electromagetic Acoustic Resonators

Before moving on to alternative devices realizing different interactions of mechanically vibrating structures with a surrounding liquid, a device shall be mentioned that is quite similar to the thickness shear mode resonators discussed above. The main difference is that no piezoelectric material is required to excite the mechanical vibrations but Lorentz forces, i.e. the forces on conductors carrying electric currents in (in our case static) magnetic fields, are utilized, which is also the link to the devices discussed below.

In particular, electrically conducting disks (and also suspended membranes) have been considered in our recent work. By arranging the disk in the vicinity of a planar coil being excited by an AC current, eddy currents are generated in the disk. Due to the static field generated by a permanent magnet placed next to the disk, Lorentz forces come into action. Depending on the geometry of the arrangement (in particular the winding pattern of the coil and the direction of the magnetic field) various mechanical vibration modes can be excited in the disk. If the disk is placed in a liquid, interaction with the liquid will occur. Exciting a dominantly shear-polarized mode, this interaction will be similar to that of TSM resonators [19].

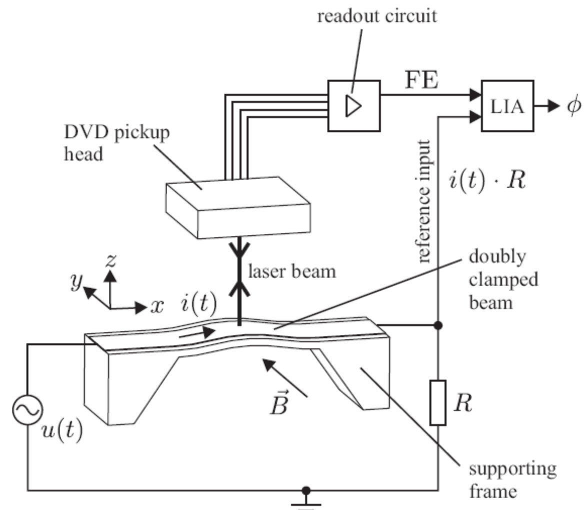
The readout can be performed by measuring the impedance of the primary coil. In case of mechanical resonances, motional inductance causes significant electric fields in the disk, which in turn generate secondary currents. These currents induce voltages in the primary coil resulting in resonance peaks which are superimposed to the base impedance of the coil.

All kinds of resonant modes can occur in the disk. The ones with significant compressional components can also be employed for liquid level sensing by utilizing interferences of the resulting standing wave between liquid level and disk surface. The latter again are represented in the impedance of the primary coil [20].

IV. Vibrating Bridge Device

A. Device Principle and Operation

In order to obtain an interaction of the liquid with the vibrating structure which is more comparable to that occurring in conventional laboratory viscometers, we considered vibrations at lower frequencies (in the kHz regime) and employing an interaction beyond pure shear vibrations. Such an interaction can be obtained by placing a vibrating bridge in the liquid. (Similar approaches and considerations involving cantilevers vibrating in liquids are discussed, e.g., in [21],[22])



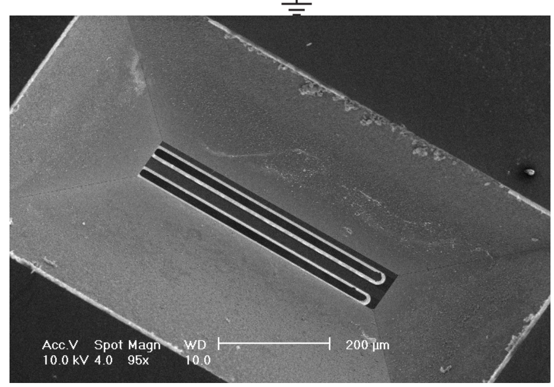


Fig. 3. Top: A micromachined vibrating bridge exited by Lorentz-forces is placed in a liquid. The readout is performed optically using a DVD-pickup unit. Bottom: SEM picture of the device (backside view showing also slanted cavity walls associated with KOH etching in the bulk micromachining process.

Fig. 3 shows such the scheme of such a device [23]. The bridge features a conductive path carrying an AC current. A permanent magnetic field is arranged such that the resulting Lorentz-forces generate lateral vibrations of the bridge. The device reported in [23] was fabricated using a micromachining process yielding a silicon-nitride (Si3N4) bridge (dimensions t=1.3 µm, l=320 µm, w=40 µm) with a fundamental resonance frequency of about 20kHz. The readout is obtained using the focusing mechanism of a commercial DVD pickup, which allows obtaining the frequency response of the device.

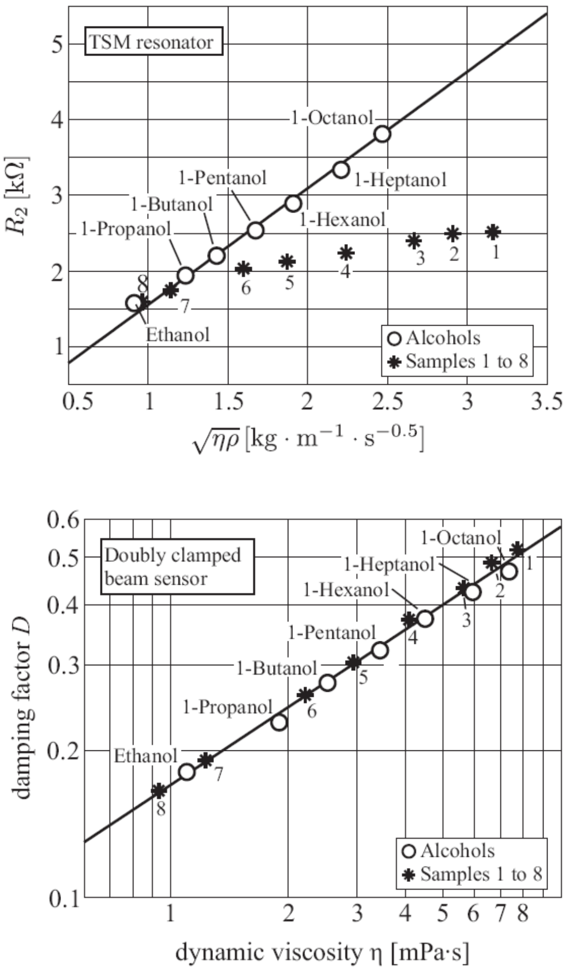


Fig. 4. Viscosity sensing of Newtonian and non-Newtonian liquids (alcohols and suspensions, respectively). Results for a TSM resonator (relation of loss resistance to $(\eta\rho)^{1/2}$, top) and bridge device (relation of damping factor to η, bottom). The bridge device gives results comparable to the laboratory viscometer also for the non-Newtonian liquids.

Fitting a second order system to the frequency response, the damping coefficient D associated with the second order system can be correlated with the viscosity of the surrounding liquid, whereas the changes in resonance frequency are dominantly correlated with the liquid's density. In [23] the viscosity sensing of a series of alcohols (considered Newtonian) and suspensions of SiO_2 particles in water (considered non-Newtonian) have been compared for a 6 MHz TSM resonator and a 20 kHz bridge resonator, see Fig. 4. For the TSM device, the loss resistance is plotted vs. the square root of the viscosity-density product (which should be linearly related to each other for Newtonian liquids) and for the bridge device the damping factor is plotted versus the viscosity. In both cases the viscosity given on the x-axis is that obtained by a laboratory viscometer (plate-cone device). It can be seen that for the bridge device the correlation is maintained, whereas for the TSM device a deviation occurs for the non-Newtonian samples. Thus, the bridge device is able to yield results comparable to that of laboratory viscometers, which can be mainly attributed to the lower vibration frequency.

A similar device has also been fabricated using plastics technology where the bridge has been obtained by structuring a polymer foil coated with a metal film using photolithography and etching technology. In this case a second conductive path has been implemented on the bridge allowing for readout by means of the induced voltage in said path [24].

B. Device Modeling

The interaction with the liquid is much more complicated than in case of TSM resonators. Nevertheless, a semi-analytical model could be utilized. The vibration of the beam can be described by using the well-known Euler-Bernoulli beam theory, where the interaction with the liquid is considered in terms of an additional distributed force term acting on the vibrating beam. Assuming harmonic vibrations and the common complex notation, this interaction force can be approximately set proportional to the local displacement, where the coefficient is complex valued and frequency dependent (allowing, e.g., to include proportionality to velocity and acceleration by means of a linear and quadratic dependence on the frequency, respectively). This coefficient can be obtained by assuming that the major flow around the vibrating beam occurs in the transverse plane (i.e. the plane whose surface normal is in the direction of the unperturbed beam axis). This allows using a 2D approximation, where a rectangular cross section harmonically vibrating in a viscous liquid is considered. Linearizing the Navier-Stokes equations, again a spectral domain/MoM approach similar to that described earlier is used to obtain the force distribution on the beam which maintains the harmonic vibration at a given amplitude. Relating the averaged force across the beam to the vibration amplitude yields the complex coefficient required for the Euler Bernoulli equation. The approach is described in detail in [25].

V. Double Membrane Device

A. Device Principle and Operation

This device uses a flat, rectangular liquid chamber (which, by featuring an inlet and an outlet channel, may be part of miniaturized fluidic system) whose top and bottom planes are embodied by elastic membranes. For the prototype device, the membranes are constructed by polymer foils which are clamped on a structured PCB-frame creating the side-walls of the liquid chamber. The membranes carry conductive paths for electromagnetic excitation and inductive readout in combination with static magnetic field provided by a permanent magnet arranged next to the chamber. Fig. 5 shows an exploded view of the prototype device (device dimensions are in the order of millimeters) [26]. Assuming approximately linear flow fields, possible motions can be represented as a superposition of the antisymmetric and symmetric membrane motions.

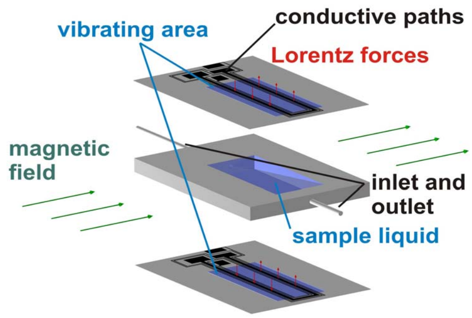


Fig. 5. Exploded view of the double membrane device.

Also for this device, the readout is obtained by recording the frequency response, which can be represented by two major parameters, i.e. resonance frequency and damping. In case of this device, the relation between the parameter pairs viscosity/density and damping/resonance frequency is more involved as in case of the bridge device since both parameters of one pair significantly influence both of the other pair as can be seen in the sample results shown in Fig. 6 [26].

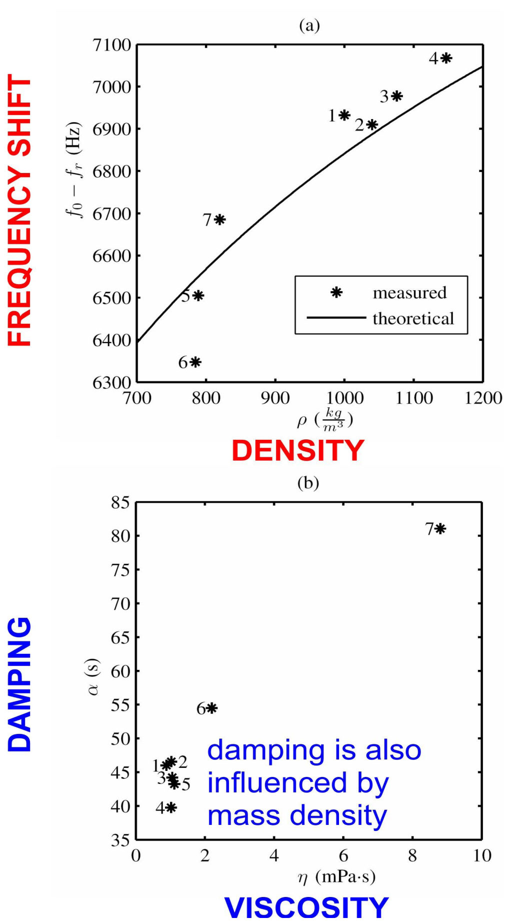


Fig. 6. For the double membrane device the relation viscosity/density and damping/resonance frequency is more involved.

B. Device Modeling

Again, modeling was attempted using at least semi-analytical approaches. This is once more motivated by the occurrence of strongly varying characteristic length scales hampering a computationally efficient implementation of the FEM.

The first approach yields only limited accuracies in the order of 10% but provides a basic idea on the relations between the above mentioned parameter pairs. It relies on the approximation of the flow field for the resonating modes by first determining the mode shape of the elastic membranes vibrating in air. It is then assumed that this basic mode shape is also present in case of liquid being filled into the chamber (of course at a very different resonance frequency). The associated flow is then approximated by solving the potential flow problem (i.e. the flow of an ideal, non-viscous liquid) using said mode shapes as boundary conditions. The resulting potential flow solution yields nonphysical nonvanishing tangential velocity components at the boundaries, which are compensated in a further step by adding damped shear waves propagating into the liquids featuring opposite shear displacements at the boundary. These shear wave contributions thus represent an approximation for the rotational flow part. The approximate total flow obtained by the superposition of these two contributions can be used to estimate the resonance frequency and the quality factor by determining the involved kinetic and potential energies as well as the viscous losses within the liquid accumulated during one cycle of the vibration. The method is described in more detail in [26].

An alternative approach that was implemented is in principle more accurate, however, this method has been implemented as a 2D approximation, which still gives the basic relations between the parameter pairs. Considering an associated 2D problem (cross section of the original problem) and linearizing the Navier-Stokes equations again, a spectral domain approach is implemented. In particular, the anti-symmetric and the symmetric case are considered, which each approximately represent a spectral line in the Fourier domain if the problem is periodically continued and sinusoidal mode shapes are assumed (this assumption can also be relaxed by considering the actually enforced Lorentz forces on the membranes). The elastic membranes and the liquid in between are considered as layers in a stratified structure. This approach is numerically very efficient and is described in [27].

VI. Micromachined Plate Device

Finally, we discuss a recently devised sensor [28]. It is based on the idea of enabling the interaction of a shear-vibrating surface with a liquid at lower frequencies than that of TSM sensors. The device consists of a micromachined Si-plate (fabricated from a SOI wafer) suspended by four beams. Across the beams conductive paths lead across the plate, which can be used to excite the device to shear vibrations in combination with a static magnetic field by a permanent magnet located next to the device. Readout is performed by piezoresistors in two of the suspension beams. Fig. 7 shows a SEM picture of the device. Due to the dominant shear interaction, the frequency response should resemble that of

TSM resonators but at lower frequencies. Fig. 8 shows the recorded frequency responses in the 10 kHz regime and the correlation of the damping factor (obtained from the frequency responses using a 2nd order system fit) with the viscosity. The device shows some spurious compressional wave excitation due to the finite plate thickness. More details on the device and the modeling can be found in [28]

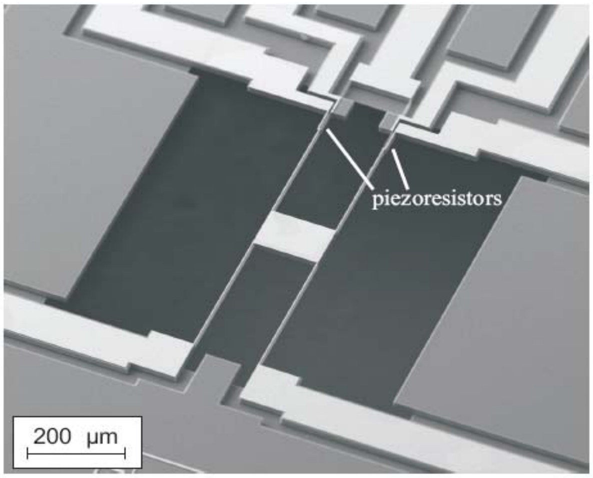


Fig. 7. Shear vibrating suspended plate in Si-micromachining technology. The plate is driven by Lorentz-forces, readout is performed by means of piezoresistors.

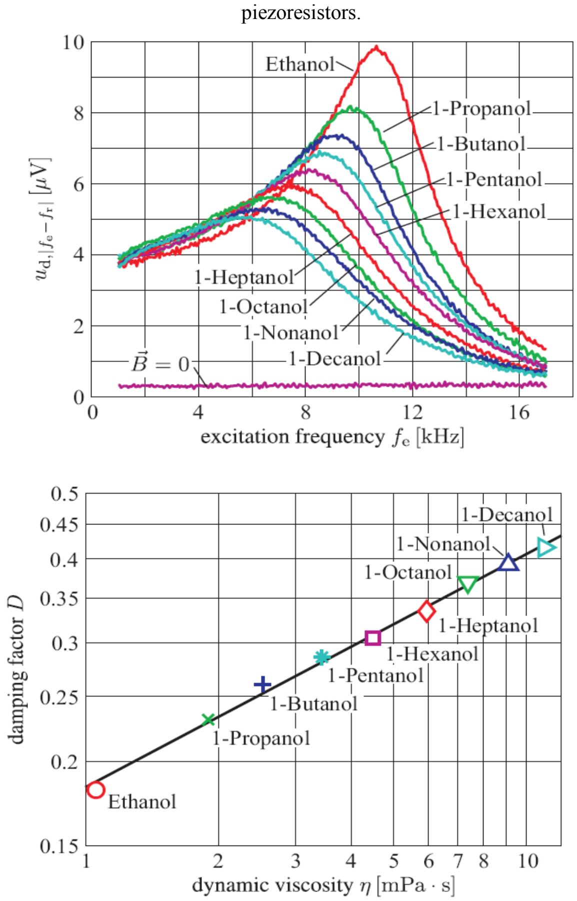


Fig. 8. Frequency response (above) and the correlation of the damping factor D with the dynamic viscosity (below) for a series of test liquids (alcohols).

VII. Summary and Conclusions

Miniaturized sensors are ideally suited for measuring the density and rheological properties (viscosity) of fluids online. Depending on the measurement method, the associated rheological domain is not necessarily comparable to that probed by established laboratory equipment. In particular, traditional shear mode sensors (e.g., TSM-resonators) measure a “thin film viscosity” at relatively high frequencies and small amplitudes, which may lead to issues when sensing complex liquids (e.g., emulsions, suspensions) since the results will in general not correlate with those obtained by laboratory instruments. Utilizing alternative devices featuring lower vibration frequencies, larger vibration amplitudes, and potentially also different vibration modes allows to sense in a domain more comparable to that of laboratory instruments. However, the interaction with the liquid is more involved leading to increased efforts in device modeling and potentially more complicated relations of the device response to the physical liquid parameters, i.e. density and viscosity.

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