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# Effect of hydrodynamic force on microcantilever vibrations: applications to liquid-phase chemical sensing

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**Abstract:**

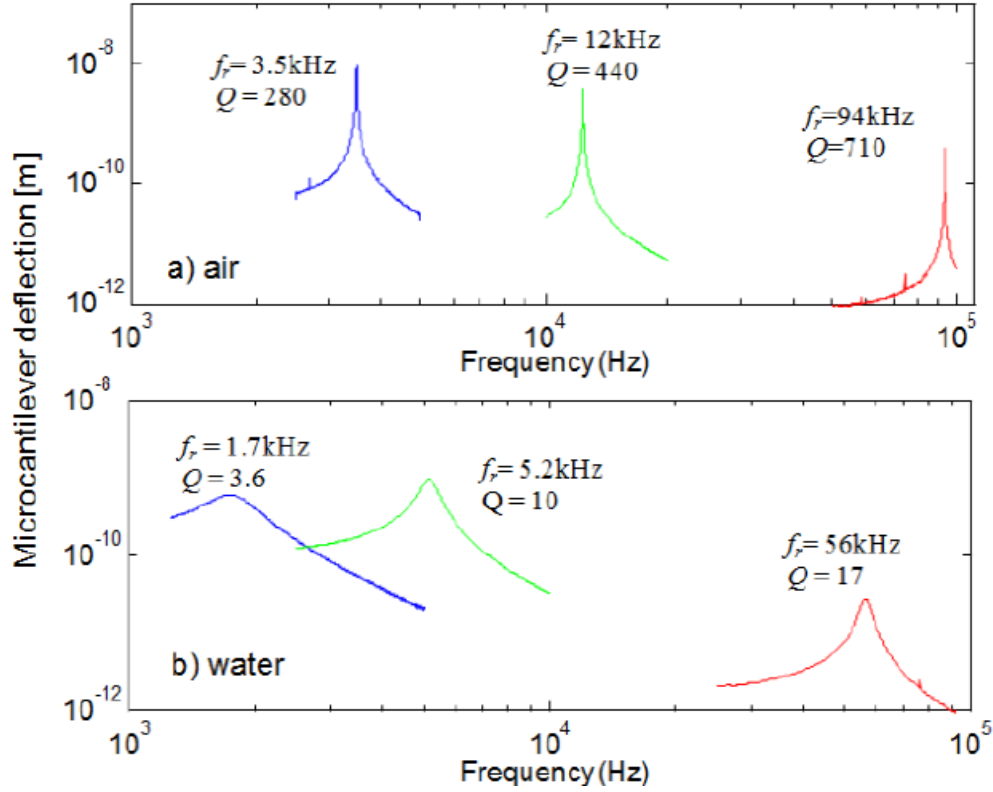
At the microscale, cantilever vibrations depend not only on the microstructure's properties and geometry but also on the properties of the surrounding medium. In fact, when a microcantilever vibrates in a fluid, the fluid offers resistance to the motion of the beam. The study of the influence of the hydrodynamic force on the microcantilever's vibrational spectrum can be used to either (1) optimize the use of microcantilevers for chemical detection in liquid media or (2) extract the mechanical properties of the fluid. The classical method for application (1) in gas is to operate the microcantilever in the dynamic transverse bending mode for chemical detection. However, the performance of microcantilevers excited in this standard out-of-plane dynamic mode drastically decreases in viscous liquid media. When immersed in liquids, in order to limit the decrease of both the resonant frequency and the quality factor, and improve sensitivity in sensing applications, alternative vibration modes that primarily shear the fluid (rather than involving motion normal to the fluid/beam interface) have been studied and tested: these include inplane vibration modes (lateral bending mode and elongation mode). For application (2), the classical method to measure the rheological properties of fluids is to use a rheometer. However, such systems require sampling (no insitu measurements) and a relatively large sample volume (a few milliliters). Moreover, the frequency range is limited to low frequencies (less than 200Hz). To overcome the limitations of this classical method, an alternative method based on the use of silicon microcantilevers is presented. The method, which is based on the use of analytical equations for the hydrodynamic force, permits the measurement of the complex shear modulus of viscoelastic fluids over a wide frequency range.

**Keywords:** Hydrodynamic force, microcantilever vibration, rheology, chemical detection, in-plane vibration, MEMS.

## 1. INTRODUCTION

In recent years, interest in microcantilever-based chemical and biochemical sensing systems has risen due to their projected high sensitivity [1-5]. The large ratio of surface area to volume makes the microcantilever extremely sensitive to surface processes. For (bio)chemical detection, the microcantilever is usually coated with a (bio)chemically sensitive layer whose purpose is to selectively sorb the analyte or molecule of interest. The sorbed substance can then be detected by monitoring either the resonant frequency shift (dynamic mode) or the quasi-static deflection (static mode). A simplified way to explain the basic principle of such sensors is to say that in the case of dynamic mode operation, the change in mass associated with the sorption of analyte onto/into the sensitive coating causes a shift in resonant frequency, which may be correlated to the ambient concentration of the target substance. For static-mode operation, the sorption of analyte causes a cantilever deflection that is induced by surface stress or by the tendency of the sensitive coating to expand or contract upon analyte sorption or desorption (modification of strain and stress in the coating). In this paper we will focus on the use of the dynamic mode.

At the microscale, cantilever vibrations depend not only on the microstructure's properties and geometry but also on the properties of the surrounding medium (density and viscoelastic properties). In fact, when a microcantilever vibrates in a fluid, the fluid offers resistance to the motion of the beam. The coupling between the structure and the surrounding viscous fluid influences the structure's mechanical resonance; in particular, the fluid causes a decrease of both the resonant frequency and the quality factor and, thus, negatively impacts the device's sensing capabilities. In gas, resonant frequencies of classical transverse flexural modes may be reduced by a few percent compared with the value in vacuum, whereas the quality factor may exhibit reductions of two orders of magnitude. This performance degradation for conventional out-of-plane flexural resonant modes is further exacerbated in liquids: the resonant frequency and quality factor values are approximately one order of magnitude smaller than their values in the gas phase (Fig. 1).



**Figure 1.** Measurements of three microcantilevers with different geometries (named in section 4 LL, LH and A respectively): 2810 x 100 x 20 $\mu$ m<sup>3</sup> (blue), 1440 x 285 x 20 $\mu$ m<sup>3</sup> (green) and 500 x 100 x 20 $\mu$ m<sup>3</sup> (red). a) spectrum in air. b) spectrum in water.

Chemical detection using microcantilever-based sensors is based on the measurement of the resonant frequency shift  $\Delta f_r$ , which is induced by the mass increase of the vibrating microstructure during sorption of the target species into the sensitive coating [6]:

$$\Delta f_r = -f_r \frac{\Delta m}{2m} \quad (1)$$

with  $f_r$  and  $m$  being the resonant frequency and mass of the microstructure, respectively. According to the partition coefficient ( $K$ ) of the analyte/sensitive coating pair [6], which is defined as the ratio of the concentration of the analyte in the coating and the ambient

concentration,  $C_A$ , of the analyte in the surrounding medium, the mass variation  $\Delta m$  is proportional to the concentration  $C_A$ . Thus, the sensitivity of the chemical sensor,  $S$ , is proportional to the resonant frequency of the microcantilever in the surrounding medium (gas or liquid) [6]:

$$S = \frac{\Delta f_r}{C_A} = -\frac{f_r K V_L}{2m} \propto f_r \quad (2)$$

with  $V_L$  defined as the volume of the sensitive layer.

The accuracy of the resonant frequency measurement depends on the sharpness of the resonant peak which is characterized by the quality factor,  $Q$ , associated with a particular resonant mode, while the noise  $N$  corresponding to the resonant frequency measurement can be considered to be inversely proportional to the quality factor [6]:

$$N \propto \frac{1}{Q} \quad (3)$$

According to Eqs. 2-3, reduced values of the resonance characteristics (resonant frequency and quality factor) in liquid media (Fig. 1) adversely affect the sensitivity and the detection limit of these types of sensors. Therefore, for liquid-phase chemical sensing applications it is imperative that the effect of the liquid on the resonant characteristics of the sensing device be understood and considered in the sensor design in order to achieve desired levels of sensing performance (sensitivity and limit of detection).

In the present paper, we propose alternative uses of microcantilevers, including the implementation of unconventional vibrational modes to overcome the aforementioned obstacles for liquid-phase operation. By understanding in detail the influence of the hydrodynamic force on the microcantilever's vibrational spectrum, the following two objectives may be achieved: (1) optimization of microcantilevers for chemical detection in liquid media and (2)

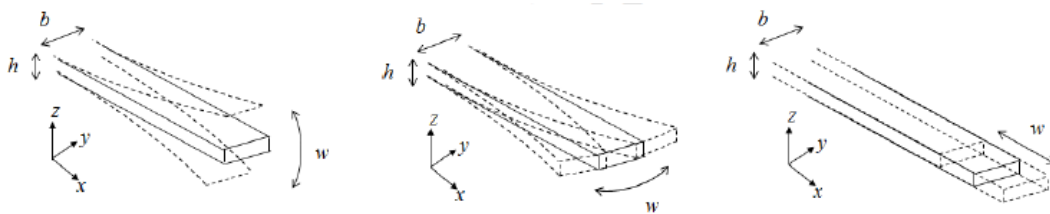
extraction of the mechanical properties of complex fluids which is of particular importance for microfluidic systems and for chemical detection in these types of environments.

The paper is organized such that a review of hydrodynamic force expressions and their impact on the resonant frequency and quality factor is given in Section 2 for the cases of out-of-plane and in-plane vibrations. In Section 3 we present how one may use the knowledge of the hydrodynamic force to choose appropriate vibrational modes and microcantilever geometries for chemical detection in liquid media. A second example of how a firm grasp of fluid-structure interaction may be used to advantage is the implementation of a microcantilever to extract the rheological properties of a fluid, which is described in Section 4.

**Notation (Fig.2):** The geometry of the cantilever is defined by the width  $b$ , thickness  $h$ , and length  $L$ . Coordinate  $x$  is measured along the beam length. The properties of the cantilever material are the Young's modulus  $E$  and the mass density  $\rho$ . The fluid (gas or liquid) properties are defined as  $\rho_f$ , the fluid's mass density,  $\eta$  the fluid's dynamic viscosity and  $G^* = G' + jG''$ , the complex shear modulus of the fluid.

## 2. INFLUENCE OF HYDRODYNAMIC FORCE ON MICROSTRUCTURE RESONANT CHARACTERISTICS

In this paper, we focus on three distinct vibration modes (Fig.2): the transverse (out-of-plane) bending mode, the lateral (in-plane) bending mode and the elongation mode (also in-plane). A fourth mode that may also have interesting potential for both chemical detection and rheological sensing is the torsional mode, whose hydrodynamic force and resonant characteristics have been studied in [7-8]; however, that mode is not included in the present study.

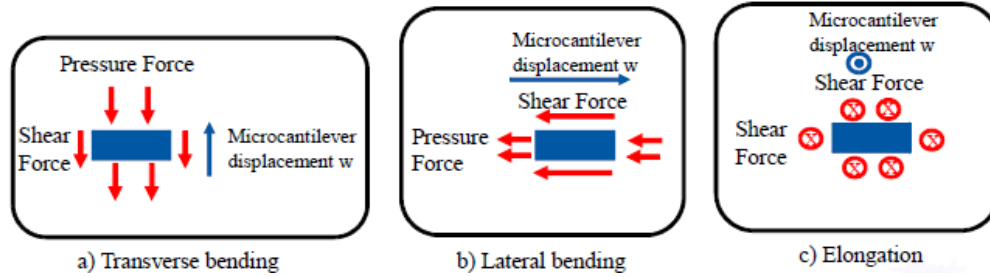


**Figure 2.** Schematic representation of the transverse (out-of-plane) bending mode, the lateral (in-plane) bending mode and the (in- plane) elongation mode.



## 2.1 General modeling of the hydrodynamic force

When a microcantilever vibrates in a viscous fluid (gas or liquid), the fluid offers resistance to the motion. Depending on the vibration mode and on the cantilever surface there are hydrodynamic pressure forces (normal to the surface) and hydrodynamic viscous forces (tangential to the surface) (Fig. 3).



**Figure 3.** Schematic representation of the hydrodynamic force on a microcantilever cross section in the cases of transverse (out- of-plane) bending, lateral (in-plane) bending and (in-plane) elongation modes.

The force per unit length,  $F_{fluid}$ , which is the consequence of all normal and tangential stresses exerted by the fluid on all the surfaces of the cantilever, can be written in the frequency domain as [9]:

$$F_{fluid} = -[j\omega g_1(x, \omega) - \omega^2 g_2(x, \omega)]w(x, \omega) \quad (4)$$

where  $x$  is the longitudinal coordinate,  $\omega$  the radial frequency of vibration,  $w$  the microcantilever deflection in the case of bending modes or the microcantilever axial displacement in the case of the elongation mode, and  $g_1$  and  $g_2$  are functions depending on the fluid properties and the microcantilever cross-sectional geometry. They may be interpreted, respectively, as the distributed damping coefficient of the fluid and the distributed effective fluid mass (per unit length of beam). In general, these quantities are both frequency-dependent and  $x$ -dependent; however, in what follows  $g_1$  and  $g_2$  will be assumed uniform in  $x$  as this assumption has been shown to be sufficiently accurate in many cases of practical interest [10].

It is common to normalize  $F_{fluid}$  into a dimensionless form called the hydrodynamic function,  $\Gamma = \Gamma_r - j\Gamma_i$ , (where  $\Gamma_r > 0$  and  $\Gamma_i > 0$ ) [11]

$$\Gamma_r = \frac{4}{\pi \rho_f b^2} g_2 \quad (5)$$

$$\Gamma_i = \frac{4}{\pi \rho_f b^2} \frac{g_1}{\omega} \quad (6)$$

The hydrodynamic function,  $\Gamma$ , represents the total hydrodynamic force per unit length normalized by the force per unit length required to excite (at the same amplitude and frequency) a circular cylindrical volume of diameter  $b$  and density  $\rho_f$ . The subscripts  $r$  and  $i$  in Eqs.5-6 denote the real and imaginary parts of the hydrodynamic function, respectively.

## 2.2 Resonant characteristics of the microstructure

The fluid effects (viscous term  $g_1$ , and inertial term  $g_2$ ) influence the dynamic response of the beam; in particular, the resonant frequency,  $f_r$ , and the quality factor associated with viscous losses,  $Q_{\text{visc}}$ , may be expressed as [10-11]

$$f_r = f_{0,vac} \frac{1}{\sqrt{1 + Lg_2/m}} \sqrt{1 - \frac{1}{2Q^2}} \quad (7)$$

$$Q_{\text{visc}} = \frac{2\pi \sqrt{1 + Lg_2/m}}{Lg_1/m} f_{0,vac} \quad (8)$$

where  $f_{0,vac}$  is the undamped natural frequency of the microcantilever in vacuum,  $Q$  is the total quality factor of the cantilever/fluid system (incorporating all losses),  $m$  is the total microcantilever mass and  $L$  is the microcantilever length.

The above equations illustrate, as stated in the introduction, the fact that in the general case of dynamic mode operation of a microcantilever, the resonant frequency and the quality factor depend on the fluid properties via the terms  $g_1$  and  $g_2$ . In the following sections, the terms  $g_1$  and  $g_2$  will be analyzed for the different vibration modes.

### 2.3 Transverse (out-of-plane) bending vibrations of thin microcantilevers

The hydrodynamic function of a transversely vibrating beam of ribbon-like cross-section (i.e., width  $b$  much larger than the thickness  $h$ ) was previously determined in [12] by using the method of moments to solve for the velocity field in the fluid and in [11] by performing a fit of the numerical results of [12].

A more accurate approximation of the total hydrodynamic force acting on a transversely vibrating microcantilever in liquids should include both the effects of the pressure and shear stress exerted by the fluid on all faces of the beam of finite cross-sectional dimensions. Due to the symmetry of the problem, the hydrodynamic forces acting in the  $x$  or  $y$  direction on a beam with a rectangular cross-section vibrating transversely will cancel each other out. Only the hydrodynamic forces acting in the  $z$  direction will then affect the response of the beam. These forces (Fig.3a) are the pressure forces acting on the large faces (of dimension  $b$ ) and the shear forces acting on the small faces (of dimension  $h$ ). It can be assumed that the microcantilever is long enough so that the hydrodynamic force acting on the surface at the unsupported end of the beam is negligible.

Recently, results for the hydrodynamic function in the case of transverse bending that accounted for both the beam's aspect ratio ( $h/b$ ) and the Reynolds number ( $Re$ ) of the fluid flow have been published [13]. A Taylor series of the analytical function or a fitting of the numerical results can be made, and then the hydrodynamic function  $\Gamma$  can be written as

$$\Gamma_r = a_0 \left(\frac{b}{h}\right) + a_1 \left(\frac{b}{h}\right) \frac{1}{\sqrt{Re}} + a_2 \left(\frac{b}{h}\right) \frac{1}{Re} \quad (9)$$

$$\Gamma_i = b_0 \left(\frac{b}{h}\right) + b_1 \left(\frac{b}{h}\right) \frac{1}{\sqrt{Re}} + b_2 \left(\frac{b}{h}\right) \frac{1}{Re} \quad (10)$$

With  $a_0, a_1, a_2, b_0, b_1$  and  $b_2$  being functions of aspect ratio  $b/h$ , and the Reynolds number,  $Re$ , defined as

$$Re = \frac{wb^2\rho_f}{4\eta} \quad (11)$$

For the ribbon-like case,  $a_0$ ,  $a_1$ ,  $a_2$ ,  $b_0$ ,  $b_1$  and  $b_2$  have constant numerical values. In [14] a fit of the results of [11] has been made and the obtained numerical values are:

$$\begin{aligned} a_0 &= 1.0553 & a_1 &= 2.6868 & a_2 &= 0 \\ b_0 &= 0 & b_1 &= 2.6883 & b_2 &= 1.3682 \end{aligned} \quad (12)$$

#### *2.4 In-plane vibrations of thin microcantilevers: lateral bending and elongation*

In 1851, Stokes investigated the forces acting on an infinite rigid plane undergoing a sinusoidal, in-plane motion [15]. This is commonly called Stokes's second problem. The fluid forces on a laterally vibrating beam of infinitely thin cross-section can be considered equivalent to those of Stokes's second problem if the beam under investigation is also quite wide relative to the boundary layer thickness of the fluid. In this case, the hydrodynamic force can be expressed as

$$\Gamma_r = \Gamma_i = \frac{4}{\pi\sqrt{2Re}} \quad (13)$$

This equation corresponds to the more general form of Eqs. 9-10 for the following values of coefficients:

$$\begin{aligned} a_0 &= 0 & a_1 &= 0.9003 & a_2 &= 0 \\ b_0 &= 0 & b_1 &= 0.9003 & b_2 &= 0 \end{aligned} \quad (14)$$

However, Eq. 13 neglects edge effects, which could be significant for vibrations in which the boundary layer thickness is not negligible relative to width  $b$ . Furthermore, it also neglects the fluid resistance due to pressure on the thin faces of the beam (those of corresponding dimension  $h$ ) in the lateral bending mode case and the fluid resistance due to shear on the same faces in the elongation mode case (Figs. 3b-3c). These neglected terms may be important for many practical beam geometries. As was the case for out-of-plane vibrations, it can be assumed that the microcantilever is long enough so that the hydrodynamic force acting on the surface at the unsupported end of the beam is negligible.

In the case of lateral bending, more accurate modeling has been studied in [13,16,17]: the work takes into account both of the edge effects, which could be significant for beams whose boundary layer thickness is not much smaller than the width  $b$ , and the fluid resistance due to pressure on the thin faces of the beam. These results may also be fit to express them in the form of Eqs. 9-10.

In the case of the elongation mode, it is easy to modify Eq. 14 in order to take into account the hydrodynamic force on the thin faces of the beam (of dimension  $h$ ):

$$\begin{aligned} a_0 &= 0 & a_1 &= 0.9003(1+h/b) & a_2 &= 0 \\ b_0 &= 0 & b_1 &= 0.9003(1+h/b) & b_2 &= 0 \end{aligned} \tag{15}$$

In this mode, if we want to take into account the edge effects, numerical simulation may be performed and fitted to express them in the form of Eqs. 9-10.

### **3. CHOICE OF VIBRATIONAL MODES AND MICROCANTILEVER GEOMETRY FOR CHEMICAL DETECTION IN LIQUID MEDIA**

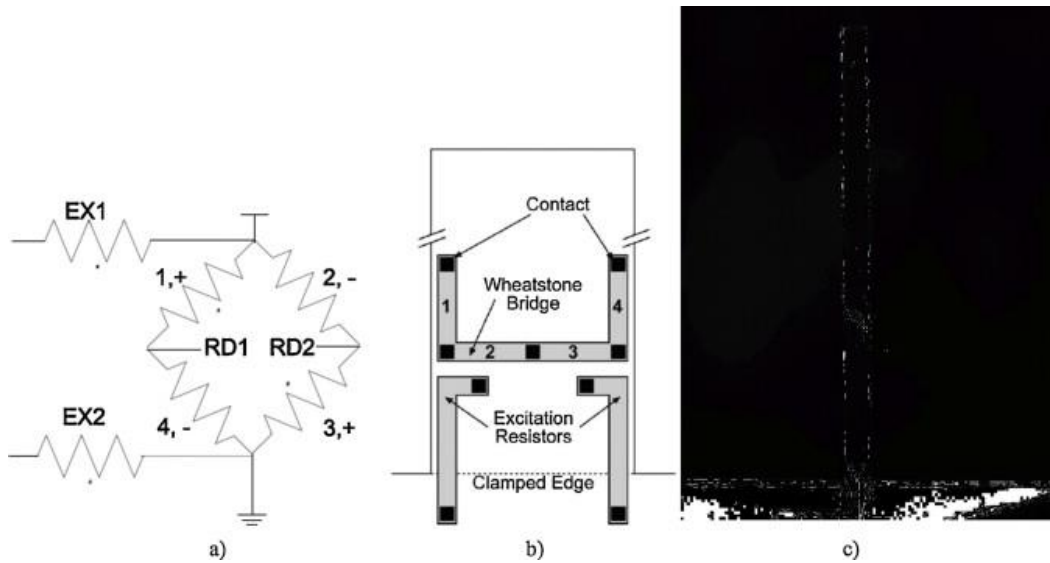
From Eq. 7, it can be seen that the decrease of the resonant frequency when the microcantilever is immersed in a fluid is essentially due to the mass effect  $g_2$ ; this effect can be minimized if the term  $g_2$  is small in liquid. On the other hand, the strong decrease in the quality factor in liquid is due to the stronger influence of the

viscous term  $g_1$  compared to the (beneficial) influence of the displaced fluid mass (term due to  $g_2$ ;) in Eq. 8.

In order to limit the decrease of both the resonant frequency and the quality factor upon immersion in liquid media, alternative vibration modes that essentially shear the surrounding fluid rather than exerting normal stress on it have been studied (section 2.4); in particular, a comparison of values listed in Eqs. 12 and 14, within the context of the expressions for the hydrodynamic function (Eqs. 5, 6, 9 and 10), indicates quantitatively that the in-plane modes result in much smaller  $g_1$  and  $g_2$  values than the out-of-plane mode, meaning that the in-plane modes are less affected by immersion in liquid. Examples of analyses and experimental measurements of the resonant frequency and quality factor associated with the two unconventional modes of lateral bending and axial elongation are presented in what follows.

### **3.1 Case of lateral (in-plane) bending vibrations**

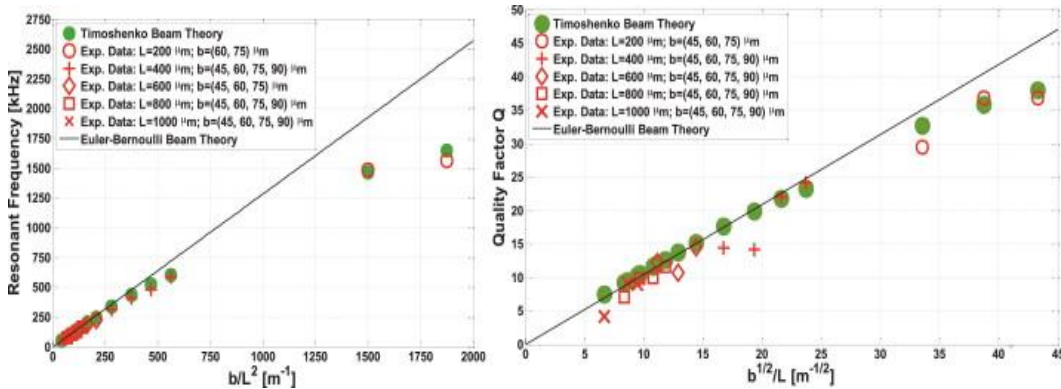
Several cantilevers with widths of 45, 60, 75 and 90  $\mu\text{m}$ , lengths of 200, 400, 600, 800, 1000  $\mu\text{m}$  and nominal silicon thicknesses of 5, 8, 12 and 20  $\mu\text{m}$ , corresponding to lateral bending resonant frequencies up to about 3 MHz, have been characterized in air and water. For operation in the lateral flexural vibration mode, thermal excitation and piezoresistive detection, both based on the use of diffused resistors, have been chosen as the actuating and sensing mechanisms, respectively (Fig. 4). The design of the actuation resistors and integrated Wheatstone bridge allows for a more efficient excitation of the in-plane flexural mode than the more classical out-of-plane flexural mode [18].



**Figure 4.** a) Schematic of the Wheatstone bridge connection, b) Schematic layout of heating resistors and piezoresistors at the microcantilever clamped-end, c) photograph of a lateral-mode silicon microcantilever with piezoresistors and heating resistors at the clamped-end.

The modeling of the fluid interaction terms,  $g_1$  and  $g_2$ , according to Eq. 13 for the lateral mode results in the resonant frequency being approximately proportional to  $b/L^2$  (where  $b$  is the cantilever width and  $L$  the cantilever length), the relative resonant frequency shift from air to water being proportional to  $L/(hb^{\frac{1}{2}})$  and the quality factor (in liquid) being proportional to  $hb^{\frac{1}{2}}/L$  [19-21]. These simple analytical results are based on elementary (“Euler-Bernoulli”) beam theory, which is applicable to slender beams (i.e., small  $b/L$  for the lateral bending case), and a Stokes-type fluid resistance model. The simple expression for  $Q$  gives excellent quantitative agreement with the measured quality factor in water for aspect ratios up to  $h/b = 0.2$  [19-21]. However, experimental results show that, for microcantilevers having larger width- to-length ratios, the behaviors predicted by this analytical model differ from measurements due to the violation of the slender beam assumption. To more accurately model microcantilever resonant behavior in viscous fluids and to improve the understanding of lateral-mode sensor performance, a new analytical model has been developed, incorporating both Stokes-type fluid resistance and “Timoshenko beam” effects (shear deformation and rotatory inertia)

[22-24]. The latter effects are neglected in Euler-Bernoulli beam theory but become more important at larger  $b/L$  ratios, which are the types of geometries that promise larger  $Q$  values in the lateral flexural mode according to the Euler-Bernoulli analytical formula for  $Q$  listed above. The results of the Timoshenko beam model [22-24], presented in Fig. 5, exhibit a departure from linearity in both resonant frequency and  $Q$  for the shorter, "stubbier" specimens, trends that are also seen in the data. Results such as these may serve a useful purpose as guides for selecting microcantilever dimensions to maximize quality factors in liquids (in order to have small noise in the resonant frequency measurement) and to minimize the liquid-induced decrease in resonant frequency as compared to that in air (in order to yield higher sensitivity).



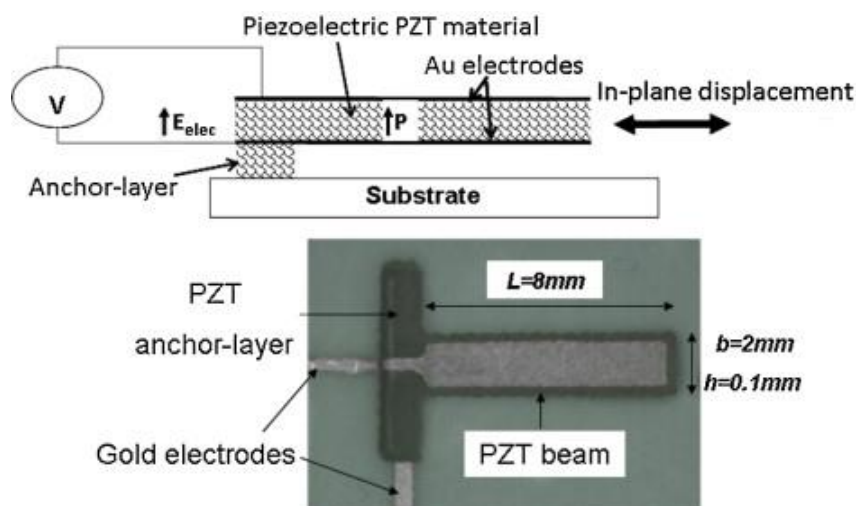
**Figure 5.** Resonant frequency and quality factor comparison (lateral mode in water,  $h=7.02 \mu\text{m}$ ,  $E=151 \text{ GPa}$ ): Timoshenko-based model, Euler-Bernoulli-based model, and experimental data [21,24].

Analyzing the experimental data in [21] shows that (1) for the first lateral flexural mode the microcantilevers exhibit quality factors up to 86 in water (for out-of-plane modes,  $Q$ -factors in water usually do not exceed 10 [25]); (2) the resonant frequencies of the first lateral flexural mode are only lowered by 3 % to 14 % in water compared to the values in air (for out-of-plane modes, this reduction is approximately 50 % [10]). These two characteristics of the lateral mode operation are promising for future sensing applications in liquid media. The first chemical detection data in liquid media generated with lateral-mode microcantilever devices are presented in [26].



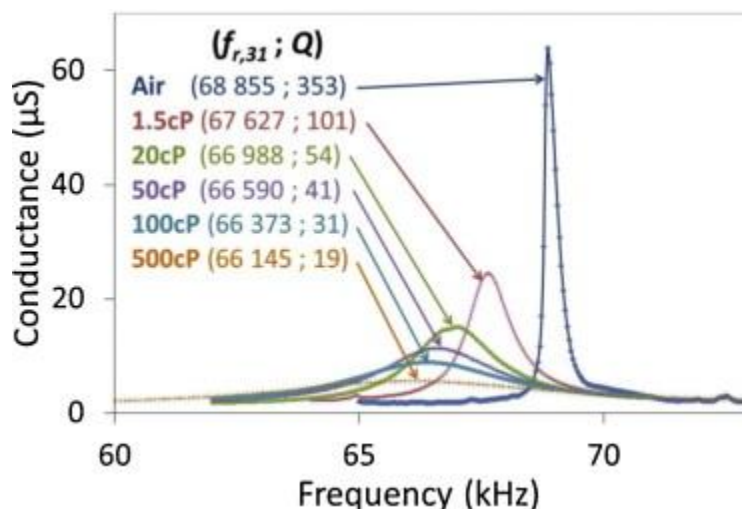
### 3.2 Case of elongation vibrations

For the same reason as for the lateral bending mode, the longitudinal mode may be of potential interest for sensing applications in liquid media. To assess the characteristics of this mode, self-actuated resonant microcantilevers based on a PZT piezoelectric thick-film sandwiched between two gold electrodes have been fabricated using screen-printing technology associated to a sacrificial layer process [27]. The microcantilevers comprising Au/PZT/Au layers [28] were partially released from an alumina substrate (Fig. 6).



**Figure 6.** Schematic of piezoelectric cantilever for elongation mode operation (top); photo of one screen-printed Au/PZT/Au microcantilever used for in-plane elongation vibrations (bottom).  $P$  represents the electrical polarization and  $E_{elec}$  the electric field.

Using an impedance analyzer (HP4194A), frequency spectra have been measured in air and in various other fluids with viscosities ranging from 1.5 to 500 cP for a piezoelectric cantilever ( $8 \times 2 \times 0.1$  mm<sup>3</sup>) with 7  $\mu$ m thick gold electrodes (Fig. 7). Even though the quality factor of 353 in air is relatively low (probably due to internal mechanical losses and piezoelectric losses), the quality factors in liquids are significantly higher compared to those of classical out-of-plane modes. The piezoelectric cantilever exhibits a decrease of the quality factor from 353 to 101 when the cantilever is brought from air to dodecane (with 1.5cP viscosity -- on the order of that of water, 1cP). At the same time, a decrease of resonant frequency of the first longitudinal mode of only 1.8% (68.8 to 67.6 kHz) is observed when the cantilever is brought from air to dodecane (1.5cP).



**Figure 7.** Effects of liquid on the resonant frequency and quality factor associated with the first elongation mode of a  $8 \times 2 \times 0.1 \text{ mm}^3$  PZT cantilever.

To demonstrate the potential advantages of the (in-plane) elongation mode in comparison with the (out-of-plane) transverse bending mode, the resonant characteristics of two cantilevers of comparable geometries in three different fluids are summarized in Table 1. The elongation-mode data corresponds to the PZT cantilever described in the previous paragraph, while the data for the transverse bending mode was obtained from measurements on a silicon cantilever of similar dimensions ( $6 \times 0.6 \times 0.11 \text{ mm}^3$ ) [29]. Despite the fact that the measurements were made on cantilevers of different dimensions and material properties, the data of Table 1 clearly suggests that the detrimental effects of the liquid on the resonant characteristics (i.e., lowering  $Q$  and  $f_r$ ), which are quite prominent in the transverse (out-of-plane) bending case, may be significantly reduced when the elongation mode is employed. This result is consistent with the earlier observations concerning lateral bending vs. transverse bending and the superiority of in-plane modes over out-of-plane modes in liquid-phase sensing applications.

As was the case with lateral-mode silicon cantilevers having an integrated scheme for actuation and measurement, the thick-film piezoelectric cantilevers actuated in the longitudinal mode are promising in terms of sensitivity and limit of detection for (bio)chemical detection in liquid media.

Liquid Viscosity	1.5 cP		20 cP		100 cP	
Mode	EL	TB	EL	TB	EL	TB
$Q$	101	19	54	4	31	2
Relative Decrease in $f_r$ (air-to-liquid)	1.8%	36%	2.7%	46%	3.6%	53%

**Table 1.** Comparison of experimental data for resonant characteristics of first elongation mode (EL) of PZT cantilever and first transverse bending mode (TB) of Si cantilever [29]. (Beam dimensions are comparable.)

## 4. USE OF HYDRODYNAMIC FORCE STUDY FOR THE MEASUREMENT OF FLUID RHEOLOGICAL PROPERTIES

Using the frequency spectra of microcantilevers vibrating in fluids and the equations of section 2, it is possible to extract the mechanical properties of fluids. As noted in the introduction and based on the results of the previous section, it is clear that successful chemical detection and analysis in liquids (Newtonian or non-Newtonian) requires that the liquid properties be accurately characterized. Different methods for fluid property determination have been developed using the transverse bending mode which, as noted earlier, is more influenced by the fluid properties than other modes [30]. The first of these permits the determination of both the mass density and viscosity of the fluid at the resonant frequency of the microcantilever in the fluid [29]; the second enables one to extract the same properties over a frequency range that includes the in-fluid resonant frequency [31]; two other methods are based on the same principle, but with the assumption that the fluid's mass density is known. These latter methods allow for the determination of the real and imaginary parts of the fluid's shear modulus, which characterize both the elasticity and viscosity of the fluid [32]. In this section we will focus on presenting a more detailed description of the fourth method (estimation of real and imaginary parts of the shear modulus over a range of frequencies).

Using the amplitude and phase frequency characteristics of the transverse-bending microcantilever, it is possible to determine the value of the hydrodynamic terms,  $g_1$  and  $g_2$ , at each measurement frequency.

Then, based on Eqs. 5-6 and 9-12, two analytical equations can be used to obtain the numerical values of both the real  $G'$  and the imaginary  $G''$  parts of the fluid shear modulus at the frequency  $f$ :

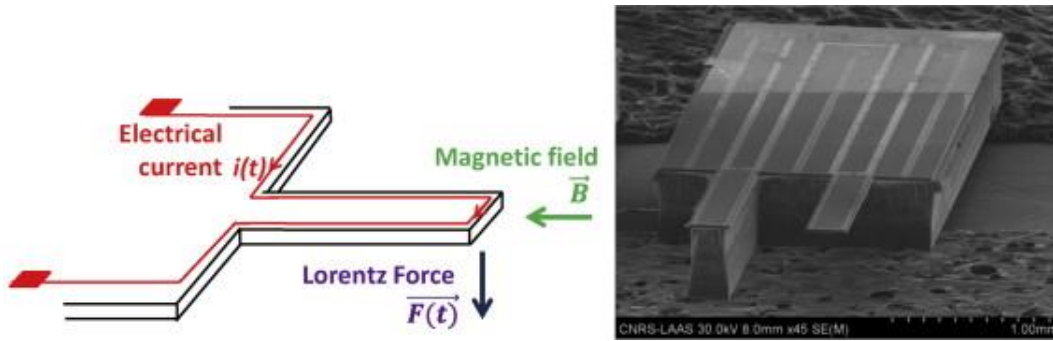
$$G'' = \frac{g_1}{D} - \frac{B}{D\sqrt{2D}} \sqrt{\left(\frac{B^2}{D} + 2(g_2\omega - C)\right)^2 + 4g_1^2 - \frac{B^2}{D} - 2(g_2\omega - C)} \quad (16)$$

$$G' = \frac{1}{D} \left( g_2\omega - C - \frac{B^2 G''}{g_1 - DG''} \right) \quad (17)$$

With

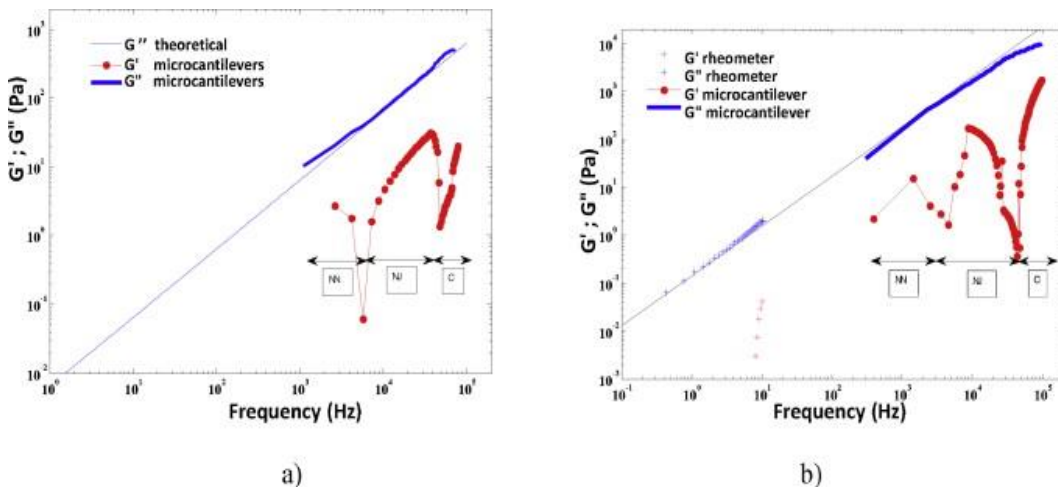
$$B = \frac{\pi b_1}{2\sqrt{2}} b \sqrt{\rho_f} \quad C = \frac{\pi}{4} \rho_f b^2 \omega \quad D = \frac{\pi b_2}{2\omega} \quad (18)$$

In order to measure the amplitude and phase transfer characteristic of microcantilevers in different fluids, chips with silicon-based microcantilevers of uniform rectangular cross section were provided by LAAS-CNRS of Toulouse, France (Fig. 8). A series of microcantilevers, termed LL, LH, and A, having the following lengths and widths were tested:  $L_{LL}=2810\mu\text{m}$ ,  $L_{LH}=1440\mu\text{m}$ ,  $L_A=500\mu\text{m}$ ,  $b_{LL}=100\mu\text{m}$ ,  $b_{LH}=285\mu\text{m}$ ,  $b_A=100\mu\text{m}$ . Due to the use of SOI wafers, the silicon thicknesses of all microcantilevers are the same,  $h = 20\mu\text{m}$ . The microcantilevers were designed to be electromagnetically actuated, i.e., excited by a Lorentz force [29]. For this purpose, a conducting path was deposited on the top surface of the microcantilever and an external magnet was used. For this method of rheological measurement, vibration amplitude measurements were made using an optical vibrometer that detects the out-of-plane velocity using Doppler interferometry (MSA 500 Polytec), after which the deflection is deduced from the velocity.



**Figure 8.** Schematic (left) and photo (right) of silicon microcantilever for operation in the transverse (out-of-plane) bending mode.

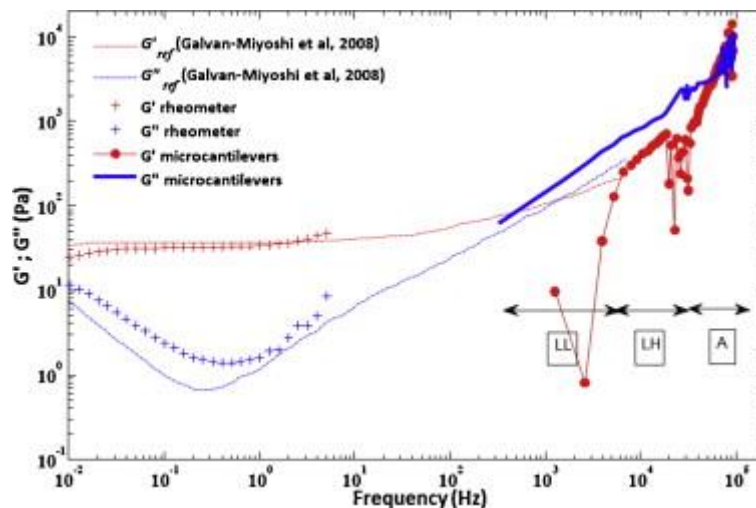
The rheograms presented in Fig. 9 illustrate the Newtonian behavior of water and of a silicone oil (20cP) obtained from spectrum measurements using three different cantilevers (LL, LH and A) covering different frequency ranges: LL between 1kHz and 5kHz, LH between 5kHz and 50kHz and A between 50kHz and 100kHz. The obtained viscous shear modulus,  $G''$ , over the whole frequency range is continuous and proportional to the frequency. This means that the viscosity of the tested fluid is constant over this frequency range and as seen in Fig. 9 it corresponds to the expected viscosity values of 1cP and 20cP. Moreover, we note that more than one decade separates the two moduli; thus,  $G'$  can be considered negligible compared to  $G''$  and the fluids can be considered Newtonian (i.e., they have no elasticity).



**Figure 9.**a) Rheogram of water (1cP) measured with three microcantilevers (LL, LH and A) and using Eqs. 16-18, b) Rheogram of silicone oil (20cP) measured with

three microcantilevers (LL, LH and A) and using Eqs. 16-18 and measured with a classical rheometer.

Some complex fluids have also been characterized using the same three microcantilever geometries (LL, LH and A). An example of characterization of CTAB (CetylTrimethylAmmonium Bromide) with microcantilevers at 19°C is given in Fig. 10, as are measurements made with the same concentration of polymer by Galvan-Miyoshi et al. at 22°C [33] and cone-plate measurements made at 19°C. As expected, unlike Newtonian fluids,  $G'$  is no longer negligible. Except for the  $G'$  measurements obtained with the LL cantilevers ( $G'_{LL}$ ), all sets of measurements of  $G'$  and  $G''$  clearly follow the same trends until 100 kHz, given the temperature difference. The inconsistent results obtained for  $G'_{LL}$  are most likely due to some cantilever inhomogeneities or to non-ideal clamping, effects which are not yet taken into account in the model. Nevertheless, except for  $G'_{LL}$ , the cantilever-based shear modulus measurements seem to corroborate and extend the Diffusion Wave Spectroscopy reference measurements [33] up to 100 kHz.



**Figure 10.** Rheogram of CTAB measured with a classical cone-plate rheometer and three cantilever geometries (LL, LH and A) and covering the frequency range 103-105 Hz using Eqs. 16-18. Comparison with literature DWS (Diffusing Wave Spectroscopy) measurements [33].

## 5. CONCLUSION

In a liquid medium, the influence of the hydrodynamic force on the microcantilever's dynamic response is much more pronounced than

in a gas medium. Appropriate modeling of this hydrodynamic force, taking into account the particular mode of vibration, is necessary in order to understand the complex interplay among the various system parameters and to achieve the promising potential of these types of devices in liquid-phase chemical sensing applications and beyond. In this paper, we have provided an overview of the relevant hydrodynamic force equations for three different modes of vibration (transverse bending, lateral bending and elongation) and presented two different applications for these modeling efforts: (a) the choice of optimal geometry and vibration mode for liquid-phase chemical detection and (b) the determination of the frequency-dependent rheological properties of liquids, which is also of paramount importance in achieving successfully detection of chemical species in more complex fluid environments.

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**Isabelle Dufour** graduated from Ecole Normale Supérieure de Cachan in 1990 and received the Ph.D. and H.D.R. degrees in engineering science from the University of Paris-Sud, Orsay, France, in 1993 and 2000, respectively. She was a CNRS research fellow from 1994 to 2007, first in Cachan working on the modelling of electrostatic actuators (micromotors, micropumps) and then after 2000 in Bordeaux working on microcantilever-based chemical sensors. She is currently Professor of electrical engineering at the University of Bordeaux and her research interests are in the areas of microcantilever-based sensors for chemical detection, rheological measurements and material characterization.

**Etienne Lemaire** got his Ph.D in 2013 from University of Bordeaux 1 in France, working on viscoelastic characterization of fluids using MEMS. His current research interest concerns the design and fabrication of low-cost sensors using natural materials.

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**Hélène Debéda** engineer in Physics of materials received the PhD degree from Bordeaux University, France, in 1996, for her work on the selectivity and reliability of thick film methane gas sensors. From 1996-1998 she obtained an European postdoc grant at the IMT Laboratory of Karlsruhe, Germany, to work on "Integrated piezoactuators made by the LIGA technique". Thanks to both the PhD and the post-doctoral position Hélène Debéda acquired a microsystem culture. She is now associate professor at the University of Bordeaux, IMS Laboratory where she is currently working on thick film technology applied to MEMS and passive components. Microstructuration and characterization of organic and inorganic free-standing thick films for gas sensing, force sensing and SHM have been recently realized.

**Claude Lucat** received his PhD in inorganic Chemistry from Bordeaux University in 1980. He joined the Centre National de la Recherche Scientifique in 1979. Currently, he is Director of Research at IMS laboratory, University of Bordeaux, France. His main research interests were in thick-film materials for hybrid components and chemical sensors. Now, his research efforts are on the fabrication and characterization of free-standing structures for Microsystems applications, using the association of screen-printing technology and sacrificial layer method specifically developed at IMS. Microcantilevers or self-supporting devices for gas sensing, force sensors, energy harvesting, etc. are studied.

**Stephen M. Heinrich** earned his BS from Penn State in 1980 and his MS and PhD degrees from the University of Illinois in 1982 and 1985, all in civil engineering. He then joined the faculty at Marquette University where he is currently Professor of Civil Engineering. Dr. Heinrich's research has focused on structural mechanics applications in microelectronics packaging and the development of new analytical models for predicting/enhancing the performance of cantilever- based chemical sensors. The work performed by Dr. Heinrich and his colleagues has resulted in over 100

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**Fabien Josse** received the License de Mathématiques et Physique in 1976, the MS and PhD degrees in Electrical Engineering from the University of Maine in 1979 and 1982, respectively. He has been with Marquette University, Milwaukee, WI, since 1982 and is currently professor in the Department of Electrical and Computer Engineering, and the Department of Biomedical Engineering. His research interests include solid state sensors, acoustic wave sensors and MEMS devices for liquid-phase biochemical sensor applications, investigation of novel sensor platforms, and smart sensor systems. Prof. Josse is a senior member of IEEE.

**Oliver Brand** received his diploma degree in Physics from Technical University Karlsruhe, Germany in 1990, and his PhD degree from ETH Zurich, Switzerland in 1994. From 1995 to 1997, he worked as a postdoctoral fellow at Georgia Tech. From 1997 to 2002, he was a lecturer at ETH Zurich, Switzerland and deputy director of the Physical Electronics Laboratory. In 2003, he joined the Electrical and Computer Engineering faculty at Georgia Institute of Technology where he is currently Professor. He has co-authored more than 150 publications in scientific journals and conference proceedings. He is a co-editor of the Wiley-VCH book series Advanced Micro and Nanosystems, a member of the editorial board of Sensors and Materials, and has served as General Co-Chair of the 2008 IEEE International Conference on Micro Electro Mechanical Systems (MEMS 2008). Dr. Brand is a senior member of the IEEE and a co-recipient the 2005 IEEE Donald G. Fink Prize Paper Award. His research interests are in the areas of silicon-based microsystems, microsensors, MEMS fabrication technologies, and microsystem packaging.