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# Viscosity–density sensor with resonant torsional paddle for direct detection in liquid

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**Abstract:** A novel micro-machined biosensor based on the resonant torsional paddle with electromagnetic excitation which can work in liquid directly is presented. The sensor designed consists of two paddles with resonant torsional mode, in which the energy loss of the resonator during the vibration is so lower that it can be suitable for detection in liquid. Finite element method analysis was carried out to guarantee the sensitivity of the sensor. Micro electro-mechanical system (MEMS) bulk silicon processes were adopted to accomplish the fabrication. A positive-feedback circuit with energy compensation is designed to improve the characteristics of the sensor in liquid. Experiments show that the resonant torsional paddle can work directly in liquid and the *Q*-factor of the sensor in liquid can be improved from 2.65 to 40 with energy compensation. Viscosity tests and density tests for the sensor show that the decrease in frequency and the decrease in *Q*-factor are related to density and viscosity of the solutions, respectively.

## 1 Introduction

With the advantages of ultrahigh sensitivity, fluorescent-labelling free, in situ detection and low-cost fabrication, the sensor based on micro-machined resonators has been attracting a great deal of attention in recent years and resonant sensors have been used in the measurement of viscosity, density, chemicals and biologics. Evoy *et al.* [1] fabricated and characterised nanopaddle in the translational and torsional modes of motion in the 90s. Etchart *et al.* [2] illuminate the relation between properties of liquid and the frequency characteristics of the resonator. Brandstetter *et al.* [3] fabricated a viscosity and density sensor, and built a model for analysing the vibrating resonator. He preliminarily verified the theory and the model. Owing to the big damping, the *Q*-value of the sensor is low and difficult for detecting the signals.

By using the torsional resonant mode, a microcantilever resonator with high *Q*-factor and high sensitivity (about 0.9 pg/Hz) in air was reported in [4]. A sensor based on the resonant torsional paddle with electromagnetic excitation is brought up in this paper. While working in torsional mode, the sensor can achieve higher *Q*-factor. The electromagnetic excitation and readout can effectively improve the signal-to-noise ratio of the sensor; moreover, the positive feedback is used to enhance the *Q*-factor of the sensor in liquid. Tests for the effects by the density and viscosity of solutions were carried out to verify the performance of the sensor in liquid.

## 2 Sensor design and fabrication

### 2.1 Principle of the sensor

The micro-machined sensor consists of two resonant torsional paddles, one paddle is used to detect the mass change resulted from liquid adhering, and the other is used as the reference to lower the noise resulted from characteristics of the liquid.

The top-view of one of the paddle is shown in Fig. 1. The paddle is clamped–clamped to the substrate through two short beams, around which the paddle can swing at its natural resonant frequency with torsional mode. Drive electrode and detect electrode are laid out along the sides of the paddle and an extra magnetic field is applied through the paddle. When AC current with the resonant torsional frequency of the paddle is applied through drive electrode, the paddle will be swinging around the beam in torsional mode. At the same time, an AC voltage with the same frequency will be induced on the both ends of the sense electrode, as shown in Fig. 1. When immersed in the liquid, the resonant frequency and the *Q*-factor will shift.

However, owing to the comparably high density and viscosity of the surrounding medium, the paddle oscillation is strongly damped in liquid, and the paddle's characteristics, such as its resonance frequency and *Q*-factor, also depend on the properties of the liquid. In order to reduce the interferences of the liquid, a reference paddle is added to the sensor. There is no liquid adhering on the surface of the reference paddle, so that the frequency

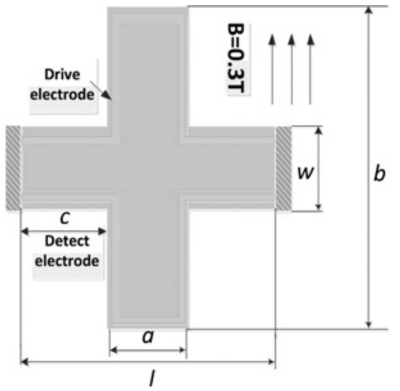


**Fig. 1** *Top-view schematic of the paddle*

shift of the reference paddle represents the property of the liquid. With the differential structure design, the performance of the sensor will be improved.

### 2.2 Structure design

The fundamental frequency of a resonator can be expressed as

$$f_t = \frac{1}{2\pi}\sqrt{K_t/I_t} \quad (1)$$

where $f_t$ is the torsional mode frequency, $K_t$ is the torsional rigidity, $K_t = 2K_l Gwh^3/c$, $I_t$ is the moment of inertia, $I_t = h(2cw^3 + ab^3)/12$, $G$ is the shear modulus, $\rho$ is the density of the paddle, $K_l$ varies with the variation of $w/h$. When $w/h > 20$, $K_l$ equals 1/3 assuming the mass variation concentrated in the paddle end.

The general expression of the frequency shift associated with an analyte uptaking on the surface of the paddle can be expressed as a function of the paddle mass variation, $\Delta m = \rho_{\text{liquid}}abn$, so (1) can be written as

$$f_t + \Delta f_t = (2\pi)^{-1}\sqrt{k_t/(I_t + \rho_{\text{liquid}}anb^3/4)} \quad (2)$$

$\rho_{\text{liquid}}$ is the density of the liquid, $a$ and $b$ is shown in Fig. 1, $n$ is the thickness of the liquid adhering to the paddle surface. Compared to the paddle size, $n$ can be neglected. So the sensor density sensitivity can be written as

$$\Delta f_t/\rho_{\text{liquid}} = (2\pi)^{-1}(54K_l Gawb^6/c)^{1/2}[\rho(2cw^3 + ab^3)]^{-3/2}n \quad (3)$$

From (3), it is known that sensitivity is irrelevant with $h$, the thickness of torsional paddle, and it decreases with the increase of $a$ and $c$. According to (3), numerical calculations were carried out to obtain the optimal parameters to achieve the better sensitivity of the paddle. Fig. 2 shows the sensitivity curve of the sensor with the optimal parameters (where $b$ is 550 μm, $w$ is120 μm, $l$ is 420 μm, $c$ is 150 μm, $h$ is 5 μm and $a$ is 120 μm). The natural torsional frequency of the paddle is 70.031 kHz and the sensitivity of the sensor is about 0.16 Hz/pg.

### 2.3 Fabrication

The paddle adopted a bulk silicon MEMS processes which used the low-stressed SiNx process on single-crystal silicon and alkaline process to release the main structure.

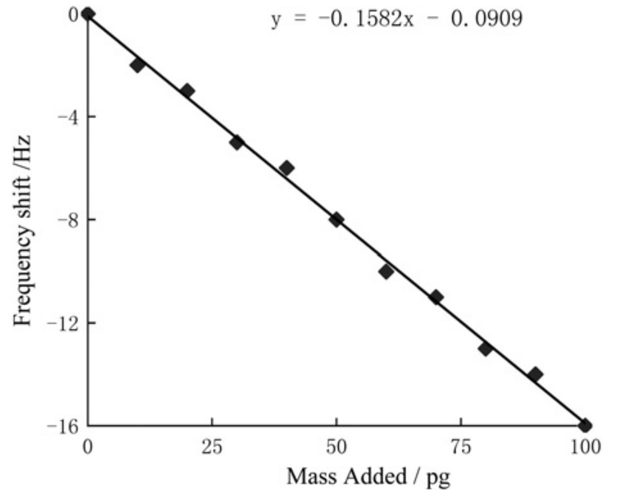


**Fig. 2** *Sensitivity of the sensor by numerical calculations with (3)*

The low-stressed SiNx layer with 5 μm in thickness by low-pressure chemical vapour deposition (LPCVD) is used as the paddle of the sensor. The main procedure is shown in Fig. 3. The processes start with the growth of a 0.2 μm thick thermal $SiO_2$ layer on the silicon substrate to compensate the stress during the deposition of the SiNx layer. Then the SiNx layer with 5 μm in thickness is deposited by LPCVD. The photoresist is patterned to define the electrode on the front of the wafer and then the Cr/Au electrodes are deposited on the SiNx layer. As the insulation of the electrodes in liquid as concerned, SiNx with 1 μm in thickness is deposited on the electrodes by plasma-enhanced chemical vapour deposition. Through dry etching SiNx by reactive ion etching, the electrode pad and

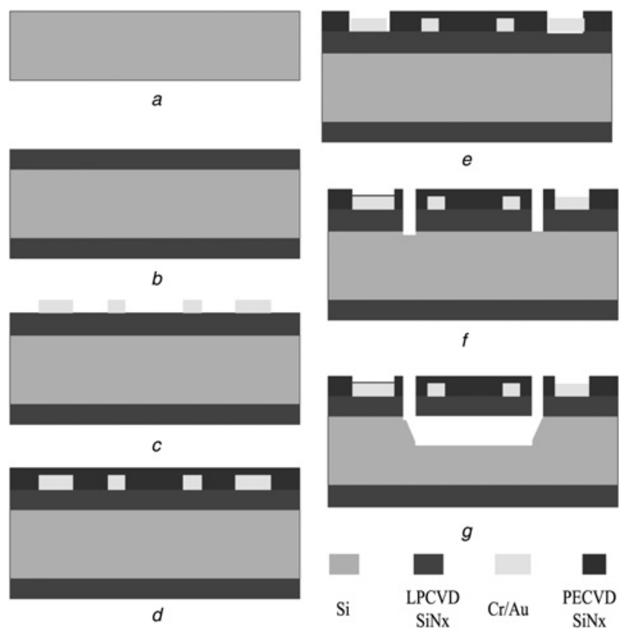


**Fig. 3** *Fabrication process schedule of the sensor*

*a* Silicon wafer cleaning
*b* SiNx deposition by LPCVD
*c* Cr/Au sputter
*d* SiNx deposition by PECVE
*e* SiNx etching by RIE to expose the electrode pad
*f* SiNx etching by RIE to obtain the process holes
*g* Wet etching to release the paddle

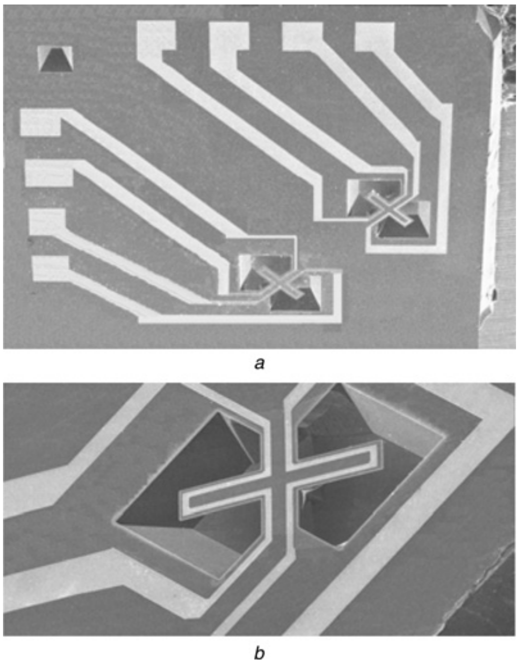

**Fig. 4** *SEM photos*

*a* Sensor
*b* Paddle

the process holes can be obtained. Finally, the paddle can be released by wet etching with alkaline solution.

Fig. 4*a* shows the scanning electron microscope (SEM) photo of the sensor, in which there are two paddles with the same structural parameters. Fig. 4*b* shows the SEM photo of the paddle in details. The sensor chip and a SmCo permanent magnet setup are assembled together and then packaged on a printed circuit board. The SmCo permanent magnet could create about 0.1 T magnetic field with in-plane direction, and the magnetic field of SmCo magnets is stable with respect to temperature change.

## 3 Circuitry

Owing to the high density and viscosity of the surrounding liquid, the $Q$-factor of the paddle will be greatly decayed and the sensitivity of the sensor will be affected obviously. So a positive-feedback circuitry was adopted to enhance the $Q$-factor of the sensor in liquid.

### 3.1 Theoritical ananlysis for energy compensation

According to the vibration theory, the paddle designed can be equivalent to the mass–spring–damp system, so the vibration function of the paddle can be expressed as

$$I_{\text{eff}} d^2\theta(t)/\text{d}t^2 + c\,d\theta(t)/\text{d}t + k\theta(t) = T_0 \text{e}^{\text{i}\omega t} \quad (4)$$

where $I_{\text{eff}}$ is the effective moment of inertia, $c$ is the damping coefficient, $\theta$ is the torsion angle, $k$ is the torsional rigidity, $T_0\text{e}^{\text{i}wt}$ is the applied torsional moment.

The electromagnetical excitation is adopted in this paper, so the detection signal is relevant to the vibration speed of the paddle. If the detection signal is amplified and then applied to the drive electrodes of the paddle, the paddle will be driven with two torsional momenta, one is $T_0\text{e}^{\text{i}wt}$ and the other is $N\lambda\text{d}\theta/\text{d}t$. Equation (4) can be rewritten as

$$I_{\text{eff}}\text{d}^2\theta(t)/\text{d}t^2 + c\text{d}\theta(t)/\text{d}t + k\theta(t) = T_0\text{e}^{\text{i}wt} + N\lambda\text{d}\theta/\text{d}t \quad (5)$$

where $N$ is the amplification coefficient.

The second part of the torsional moment proportions to the speed of the vibrating speed of the paddle, so (5) can be rewritten by

$$I_{\text{eff}}\text{d}^2\theta(t)/\text{d}t^2 + c_{\text{eff}}\text{d}\theta(t)/\text{d}t + k\theta(t) = T_0\text{e}^{\text{i}wt} \quad (6)$$

where $c_{\text{eff}}$ is the effective damping coefficient, $c_{\text{eff}} = c - N\lambda$.

As we know that

$$Q_{\text{eff}} = m_{\text{eff}}\frac{\omega_0}{c_{\text{eff}}} \quad (7)$$

where $Q_{\text{eff}}$ is the effective quality factor.

When the torsional moment is applied to the paddle, the effective damping coefficient will reduce, so the effective quality factor of the sensor will improve from (7). Here, we call it positive-feedback drive.

### 3.2 Circuitry design

The paddles of the sensor are electromagnetically actuated by Lorentz force whereas the vibration is detected through the electromagnetically induced voltage on the detect electrode. However, owing to the comparably high density and viscosity of the surrounding medium, the paddle oscillation is strongly damped in liquid. In order to enhance the frequency response of the paddle, a positive feedback is adopted into the system [5].

## 4 Experiment and results

Characteristic tests of the sensor were carried out through dynamic signal analyser (HP3562A). The resonant frequencies and $Q$-factors for torsional mode of the paddle are tested under atmosphere and in liquid.

### 4.1 Resonant characteristics test for the paddles

The resonant frequencies and $Q$-factors for torsional mode of the paddle are tested in vacuum, air and liquid, respectively. Each experiment was repeated at least three times. The resonant frequency of the paddle in vacuum is 44.7 kHz and the $Q$-factor is 260. Fig. 5*a* shows the frequency response of the paddle in liquid. Without the help of the energy compensation circuitry, big damping in alcohol makes the resonant frequency of the paddle shift from 47.7 kHz in air to 28.49 kHz in alcohol, and the $Q$-factor decrease to about 2.65. With the energy compensation of the positive-feedback circuitry, the $Q$-factor of the sensor in liquid can be improved from 2.65 to 40.

The spectra of frequency response in alcohol, 20, 40, 60 and 80% glycerol solutions is shown in Fig. 5*b*. The resonance frequency in alcohol, 20, 40, 60 and 80% glycerol solution was, respectively, 28.49, 27.05, 26.67, 24.71 and 22.62 kHz, and the corresponding $Q$-factors were 24.55, 22.27, 19.31, 14.53 and 11.37, respectively, when excited at 50 mV. From Fig. 5*b*, it is known that the resonant frequency and the $Q$-factor of the paddle decrease with the increasing ratio of alcohol. Therefore there are affirmably relationships between the frequency response of the paddle and the characteristics of the solution such as density and viscosity.

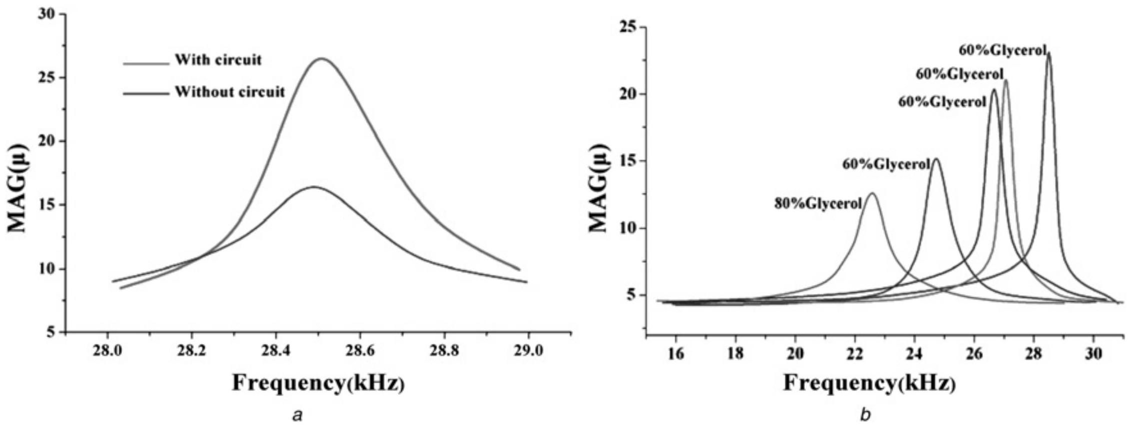


**Fig. 5** *Resonant frequency test for the paddle*

*a* Comparations of the paddle in alcohol with and without energy compensation circuit
*b* Spectra of frequency response in different solutions

### *4.2 Density measurement*

For the micro dimensions of the paddle, the Reynolds number ($Re$) under water is $9.5 \times 10^5$, and therefore inertial forces because of viscosity can be negligible [6], so the decrease in resonance frequency is primarily a function of density, and independent of viscosity [7].

As mentioned above, the inertia due to mass loading of the liquid on the sensor is responsible for the resonant frequency shift from air to liquid. Inertial forces from viscous drag of the liquid are negligible as a result of the high $Re$, the frequency response of the paddle immersed in a fluid can be expressed as [8]

$$\frac{\omega_{\text{fluid}}}{\omega_{\text{vac}}} = k\left[1 + \frac{\pi\rho a}{4\rho_c h}\right]^{-1/2} \tag{8}$$

where $\omega_{\text{fluid}}$ and $\omega_{\text{vac}}$ are the radial resonant frequencies in fluid and vacuum, $\rho$ is the density of the fluid, $a$ and $h$ are the width and thickness of the beam, $\rho_c$ is the density of the paddle, and $k$ is a constant irrelevant with paddle structure.

Density of glycerol solution is then determined from the following

$$\rho = \frac{4k\rho_c h}{\pi a}\left[\left(\frac{f_{\text{v}}}{f_{\text{f}}}\right)^2 - 1\right] \tag{9}$$

where $f_{\text{v}}$ and $f_{\text{f}}$ is resonance frequency in vacuum and glycerol solution, respectively.

The density increased monotonically with glycerol concentration. It suggests that inertial effects because of viscous drag are negligible and the frequency changes in liquid are significantly as a result of density. We can calculate the density of the liquid by (9). The results are plotted as a function of glycerol concentration in Fig. 6.

### *4.3 Viscosity measurement*

The viscous effects of the various glycerol solutions on paddle are determined by dissipative losses because of drag force of the liquid. The viscous damping coefficient $C_{\text{v}}$ can be determined by [6]

$$C_{\text{V}} = \frac{2\pi f_f (m + m_{\text{ae}})}{Q} - C_S \tag{10}$$

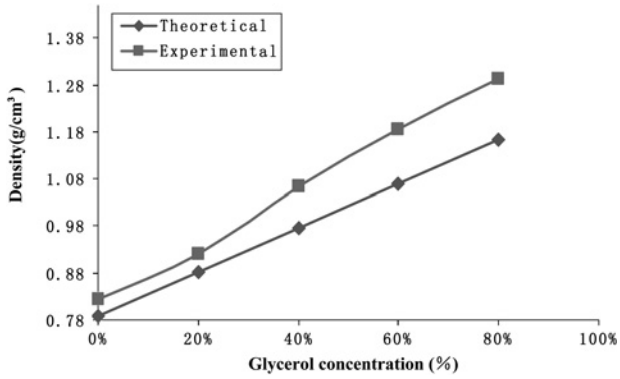


**Fig. 6** *A plot of the resonant frequency of alcohol, 20, 40, 60 and 80% glycerol solutions and the density of the solutions*

where $m$ is the intrinsic mass of the paddle, $m_{\text{ae}}$ is the added mass by the viscosity of the liquid, which can be neglected against $m$ [7], $C_s$ is the intrinsic damping coefficient, which depends on the material of construction and can be calculated by (10) with setting $C_{\text{v}} = 0$ in vacuum. Equation (10) shows a linear increase in the viscous damping coefficient with increasing glycerol concentration. The viscous effects manifest in the peak shape factor, and thus the $Q$-factors can be used to create a calibration for viscosity determination. Fig. 7 shows $Q$-factors of the paddle in

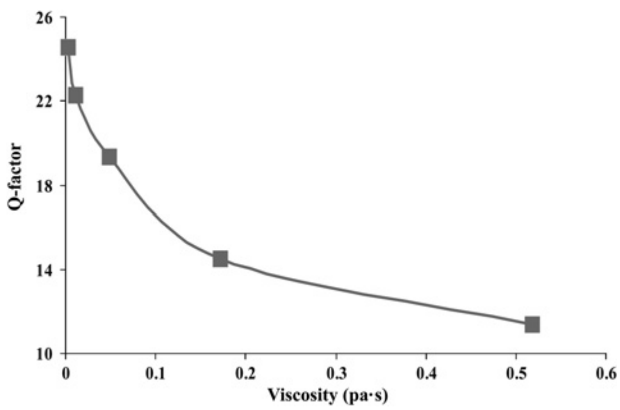


**Fig. 7** *A plot of the Q-factor of alcohol, 20, 40, 60 and 80% glycerol solutions against the viscosity of the solutions*



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solution with different viscosity, which suggest that the *Q*-factor can be used to determine the viscosity of the liquid.

## 5 Conclusion

In order to improve the sensitivity of the resonant sensor in liquid, the micro-machined resonant torsional paddle is designed and fabricated. Finite element method simulations with optimal structural parameters of the paddle show that the sensor designed is highly sensitive to the added mass in liquid (about 0.16 Hz/pg). MEMS bulk silicon processes based on Low-stressed SiNx are used to simplify the fabrications. To improve the sensitivity of the sensor, the positive-feedback circuitry is adopted with energy compensation. Characteristic tests of the sensor demonstrate that the sensor fabricated can work in liquid with better performance. The *Q*-factors of the sensor in liquid are improved to 40 with the help of the positive-feedback circuitry. The paddle sensor can be used to measure both density and viscosity from frequency response. The decrease in frequency and the decrease in peak shape factor (*Q*-factor) are related to density and viscosity of the solutions, respectively.

## 6 Acknowledgment

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## 7 References

1 Evoy, S., Carr, D.W., Sekaric, L., Olkhovets, A., Parpia, J.M., Craighead, H.G.: 'Nanofabrication and electrostatic operation of single-crystal silicon paddle oscillators', *J. Appl. Phys.*, 1999, **86**, pp. 6072–6077

2 Etchart, I., Chen, H., Dryden, P., Jundt, J., *et al.*: 'MEMS sensors for density–viscosity sensing in a low-flow microfluidic environment', *Sens. Actuators A*, 2008, **141**, pp. 266–275

3 Brandstettera, S., Riesch, C., Reichel, E.K., Jakoby, B., *et al.*: 'Sensing viscosity and density with a micromachined suspended plate resonator', *Procedia Chem.*, 2009, **1**, pp. 1467–1470

4 Jin, D., Li, X.: 'Integrated cantilever sensors with a torsional resonance mode for ultraresoluble on-the-spot bio/chemical detection', *Appl. Phys. Lett.*, 2007, **90**, p. 041901(3)

5 Tamayo, J., Humphris, A., Malloy, A., Miles, M.: 'Chemical sensors and biosensors in liquid environment based on microcantilevers with amplified quality factor', *Ultramicroscopy*, 2001, **86**, pp. 167–173

6 Kirstein, S., Mertesdorf, M., Schonhoff, M.: 'The influence of a viscous fluid on the vibration dynamics of scanning near-field optical microscopy fiber probes and atomic force microscopy cantilevers', *J. Appl. Phys.*, 1998, **84**, (4), pp. 1782–1790

7 Sader, J.E.: 'Frequency response of cantilever beams immersed in viscous fluids with applications to the atomic force microscope', *J. Appl. Phys.*, 1998, **84**, (1), pp. 64–76

8 Wilson, T.L., Campbell, G.A., Mutharasan, R.: 'Viscosity and density values from excitation level response of piezoelectric-excited cantilever sensors', *Sens. Actuators A*, 2007, **138**, pp. 44–51