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Abstract*:* The influence of the beam geometry on the quality factor and resonance frequency of resonant silicon cantilever beams vibrating in their fundamental in-plane flexural mode has been investigated in air and water. Compared to cantilevers vibrating in their out-of-plane flexural mode, utilizing the in-plane mode results in reduced damping and reduced mass loading by the surrounding fluid. Quality factors as high as 4,300 in air and 67 in water have been measured for cantilevers with a 12 μm thick silicon layer. This is in comparison to Q-factors up to 1,500 in air and up to 20 in water for cantilevers vibrating in their fundamental out-of-plane bending mode. Based on the experimental data, design guidelines are established for beam dimensions that ensure maximal Q-factors and minimal mass loading by the surrounding fluid.

Introduction

Quantification of liquid-phase analytes is essential in biomedical and environmental sample analysis. Examples of applications include, but are not limited to: (1) detection of harmful water contaminants, (2) point-of-care quantification of serum proteins, (3) chemical process monitoring, and (4) studying interactions between molecules. These four examples all require that quantitative results be obtained, preferably in real time, or with short measurement times. For the most part, these analyses are currently performed by automated, laboratory-based analytical instruments, such as mass spectrometers and UV spectrophotometers. These instruments are often expensive and complicated to operate, and thus are not suited for point-of-sampling use e.g. at a patient's beside or on-site monitoring of pollutants.

For such in-field and real-time applications, complementary metal oxide semiconductor (CMOS) compatible, cantilever-based resonators are ideal. They can readily be batch manufactured and fabricated using the same tooling as integrated circuits, which leads to reductions in cost-per-unit and also allows system-on-a-chip or system-on-a-package solutions with a small footprint [1]. Moreover, resonant sensors are of particular interest because they produce a semi-digital output signal, which can be tracked with a digital counter, thus greatly simplifying system integration. As a result, resonant cantilever sensors have been explored in recent years for a variety of (bio) chemical sensing scenarios that move in the direction of an integrated platform for point-of-sampling use [2][3][4]. To improve the limit of detection, these applications generally require resonant sensors with high Q-factors, which can be maximized by geometrical optimization. Thereby, most optimization efforts have focused on the fundamental out-of-plane flexural mode [5][6][7], which is suitable for applications in air, but is severely damped in liquids [8]. Because of the significant viscous damping in a liquid environment, most work on cantilever-based resonant chemical sensors has focused to date on gas-phase applications [2][4][9], and few studies have examined resonant sensing in the liquid phase (a notable exception being [8]). Continuous sensor operation in the fluid can avoid problems associated with washing and drying steps and also allows for measurement of the transient sensor response, which can yield additional information about the identity of an analyte.

To address this liquid phase sensing challenge, we explore the in-plane flexural mode as an alternative operation mode of cantilever sensors exhibiting (i) reduced viscous damping and (ii) reduced effective mass loading by the surrounding fluid. Recently, we have demonstrated ppb-level limits of detection for volatile organic compounds (VOC) in water using polymer-coated cantilevers operated in closed loop in liquid [10]. In the present work, we investigate the effect of the cantilever dimensions on the fundamental in-plane flexural resonance frequency and its quality factor in air and in water with the goal of establishing design guidelines for future sensor development.

The cantilever damping and mass-loading by the surrounding fluid are of particular interest because both directly affect the resonant sensor's limit of detection, which can be written as three times the smallest detectable frequency change (given by the short-term frequency stability in the case of a resonant sensor operated in closed-loop) divided by the sensor sensitivity. The short-term frequency stability is generally improved by improving the quality factor of the resonance; the sensor sensitivity of a resonant mass sensor, on the other hand, is inversely proportional to the starting mass, thus less mass addition by the surrounding fluid improves the sensor sensitivity.

Materials and Methods

A. Cantilever Fabrication and Packaging

Silicon cantilevers (Fig. 1) with lengths and widths ranging from 200–1000 μm and 45–90 μm, respectively, were fabricated and tested in both air and water. The cantilevers have integrated silicon resistors (Fig. 1) for electrothermal excitation and piezoresistive detection of transverse vibrations, whereby the resistor arrangement promotes operation in the fundamental in-plane (or strong-axis) flexural mode [11].

The beams were fabricated using a silicon bulk micromachining process that has been described elsewhere [12]. The fabrication process is based on epi-wafers with the thickness of the n-type epitaxial layer controlling the thickness of the silicon cantilevers; in the present work cantilevers with a silicon thickness of approx. 8 and 12μm have been investigated. The surface of the silicon cantilevers is covered with a 0.7μm thick thermal oxide for electrical isolation of the metal lines and a 1.2μm thick passivation stack of oxide and nitride films deposited by plasma enhanced chemical vapor deposition (PECVD) to prevent corrosion of the aluminum metallization.

The mask set used for fabrication has eight cantilevers per chip arranged around a single micromachined opening. For the present work, dies with cantilevers of the same width but different length have been tested. After dicing, each die was imaged in an SEM to determine the exact cantilever thickness. After fabrication and thickness verification, the cantilevers were die- and wire-bonded into ceramic DIL packages with acrylic inserts and a ring was glued to the surface of the die [11]. The packaging scheme allows for mechanical characterization of the beams in both air and water.

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B. Testing

For mechanical characterization in air, the frequency transfer characteristic was measured using a custom printed circuit board and an Agilent 4395A network/spectrum analyzer. The data was transferred to a computer for further processing using a LabVIEW (Austin, Texas) program. For liquid-phase mechanical characterization, a drop of deionized water was placed on the top of the die and allowed to percolate through the bulk-micromachined opening (the ring glued to the die surface prevents water from wetting the bond pads). Subsequently, the frequency transfer characteristic was captured using the same method employed in air. After data collection, any capacitive and thermal crosstalk in the frequency transfer characteristic was removed using Nyquist's method in MATLAB (Mathworks, Natick MA). The resonance frequency and quality factor were automatically extracted in MATLAB, the latter from the 3-dB width of the amplitude transfer characteristic.

Results

A. Frequency of Fundamental In-Plane Resonance Mode

The fundamental in-plane resonance frequency of the tested cantilevers ranges from 60 kHz to 2 MHz depending on the cantilever dimensions. Fig. 2 shows the measured in-plane resonance frequencies as a function of the width to length-squared ratio $\mathbf{W/L^2}$. Assuming pure beam bending, one would expect a linear increase of f with $\mathbf{W/L^2}$ (dotted line in Fig. 2).

The experimental results deviate considerably from this linear behavior for two reasons: (i) short and wide cantilevers exhibit shear deformation in addition to bending deformation and (ii) the micromachined silicon support structure is compliant compared to an ideal clamped boundary. This has been verified by finite element modeling (FEM) using COMSOL (Stockholm, Sweden). In Fig. 2, the dashed-dotted and dashed lines represent the simulated frequencies for ideally clamped cantilevers and cantilevers with a realistic silicon support structure, respectively. The silicon support structure includes

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the micromachined etch cavity and a 20μm wide silicon rim (see Fig. 3).

Compared to resonant cantilevers vibrating in their conventional out-of-plane flexural mode, operation in the in-plane flexural mode has the advantage of a reduced mass loading by the surrounding fluid. To demonstrate this, Fig. 4 shows the relative frequency change of the cantilever when immersing it in water, i.e. $(f_{air}-f_{water})/f_{air}$, as a function of the in-plane frequency in air. The observed frequency change is caused by mass loading from the surrounding water and can be minimized to values around 5% (compared to values up to 50% for the out-of-plane bending mode [8]) by using shorter and especially wider cantilevers.

B. Quality Factor of In-Plane Flexural Mode in Air

Fig. 5 shows the Q-factor of the fundamental in-plane flexural mode in air as a function of the length-to-width (L/W) ratio for 12μm thick cantilevers. Similar to cantilevers vibrating in the out-of-plane bending mode [5], an optimal L/W ratio appears to exist, with air damping reducing Q for larger L/W and support loss reducing Q for smaller L/W.

The highest measured quality factor of 4,300 in air was obtained for a 400×90×12 μm beam, i.e. for L/W≈5. The measured Q-factors in air are approx. a factor of 3 larger than the ones for cantilevers with similar dimensions vibrating in the out-of-plane mode [5]. The reason why certain cantilevers around the "optimal" L/W ratio exhibit distinctly lower Q is not understood yet and subject to further investigation; possibly, a close-by resonance mode lowers the measured Q for these cantilevers.

Fig. 6 shows the dependence of Q in air on the resonance frequency for two different cantilever widths (for various L values), again limited by air damping and support loss. The 90μm wide devices reach a regime where they are support loss limited, which is why Q drops dramatically for the shortest, i.e. highest frequency beam. The 45μm wide cantilevers, on the other hand, never reach the support loss limited region, and thus maintain high Q at the higher

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frequencies. Thus, in particular 45*μ*m wide cantilevers with in-plane resonance frequencies in the low MHz range are expected to perform well as gas-phase chemical sensors.

C. Quality Factor of In-Plane Flexural Mode in Water

Finally, Fig. 7 shows the Q-factor of the fundamental in-plane mode in water as a function of the square-root of the resonance frequency. As expected, if viscous shear forces are dominating the damping, Q increases proportional to f0.5 [13]. The highest value of 67 corresponds to a 200×90×12*μ*m cantilever. In contrast to the Q-factor data obtained in air, support loss plays no significant role in water for the range of cantilever dimensions investigated in this work, simply because the fluid damping is too high. Thus, highest Q-factors in water are obtained for the shortest, widest and thickest cantilevers. Somewhat surprisingly, the measured Q-factors in water are only slightly larger for the cantilevers with 12*μ*m silicon thickness compared to the ones with 8*μ*m silicon thickness, despite the significantly increased beam inertia. It is believed that for thicker cantilevers pressure effects on the smaller beam faces become more important and can no longer be neglected compared to the damping stemming from the larger beam faces.

Conclusions

Mechanical characterization data for the first in-plane flexural resonance mode have been presented for cantilevers with two different thicknesses and various length and width combinations. The data show that geometrical optimization can significantly improve resonator characteristics in both air and water and highlight the possibility of using resonant cantilever sensors in liquid-phase point-of-care or point-of-sampling sensor platforms. In addition, it can be seen from the data that the best cantilever dimensions for operation in air are not necessarily optimal for operation in water.

Acknowledgment

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Figures

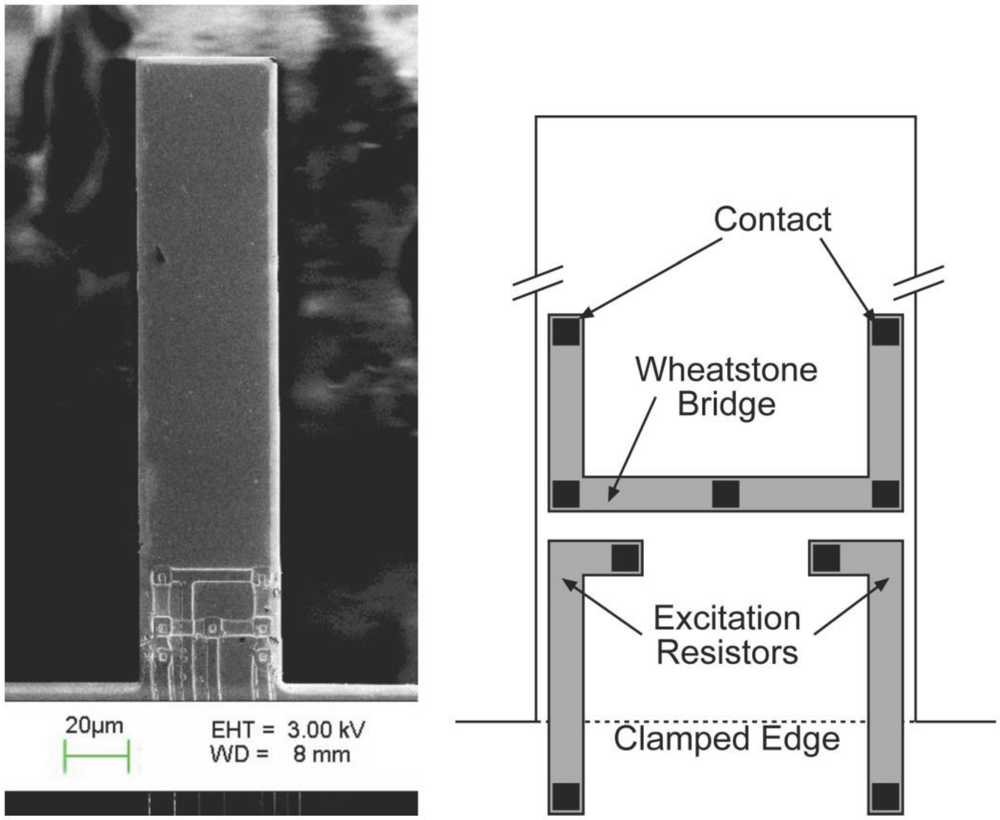

Figure 1. SEM image of a fabricated 45μm wide and 200μm long cantilever (left); schematic layout of the resistors used for thermal excitation and piezoresistive detection (right)

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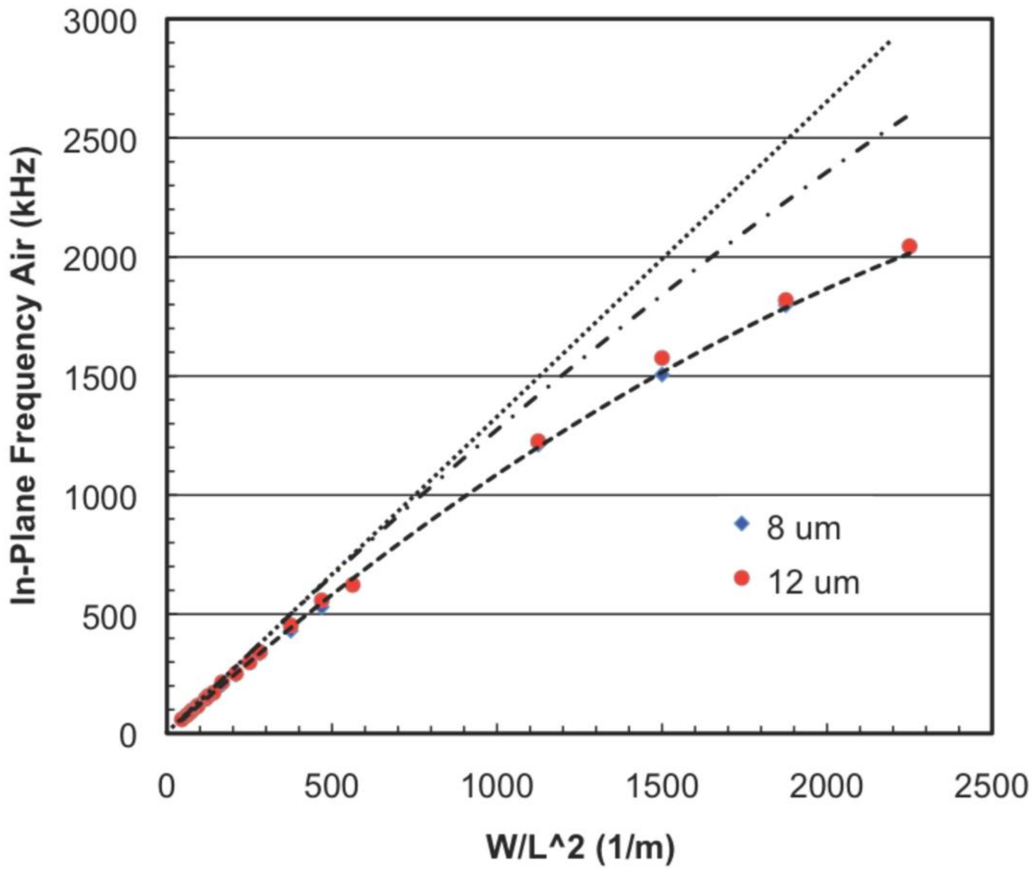

Figure 2. In-plane resonance frequency in air as a function of the width to length-squared ratio $\mathbf{W/L^2}$; symbols represent experimental data for cantilevers with silicon thicknesses of 8 and 12μm. The dashed-dotted line represents FEM simulation results for an ideally clamped cantilever, the dashed line represents FEM simulation results for cantilevers with a silicon support structure as shown in Fig. 3; the dotted line is the linear relationship expected in the case of pure bending

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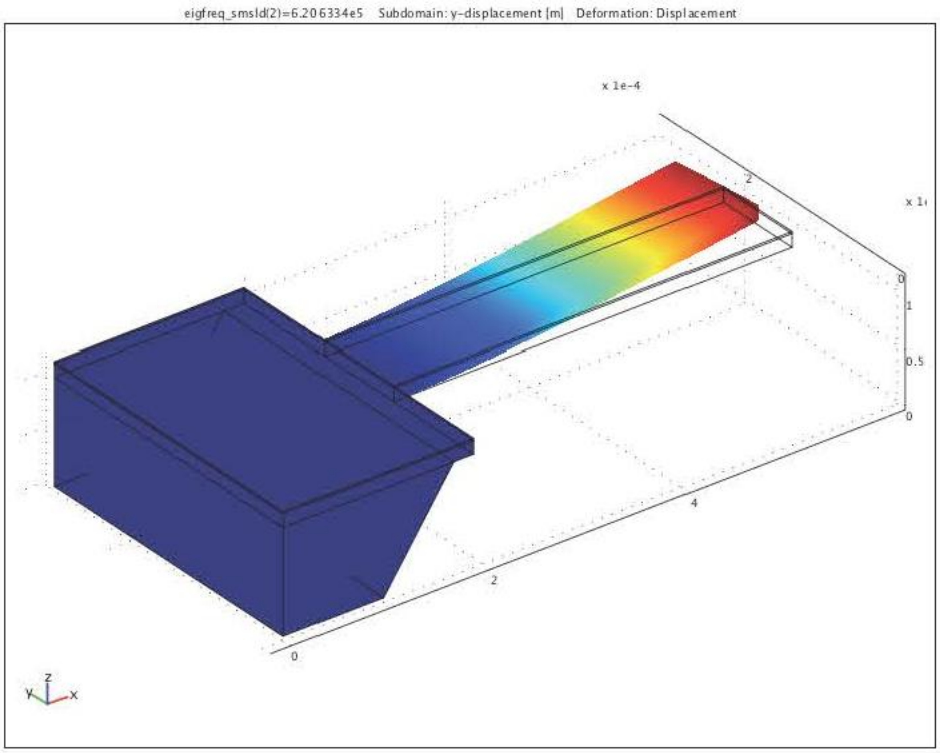

Figure 3. FEM model of a 400×90×12 μm cantilever with silicon support structure and 20μm wide silicon rim surrounding the anisotropically etched cavity. The color coding represents the in-plane (y-axis) displacement of the beam in the fundamental in-plane resonance mode

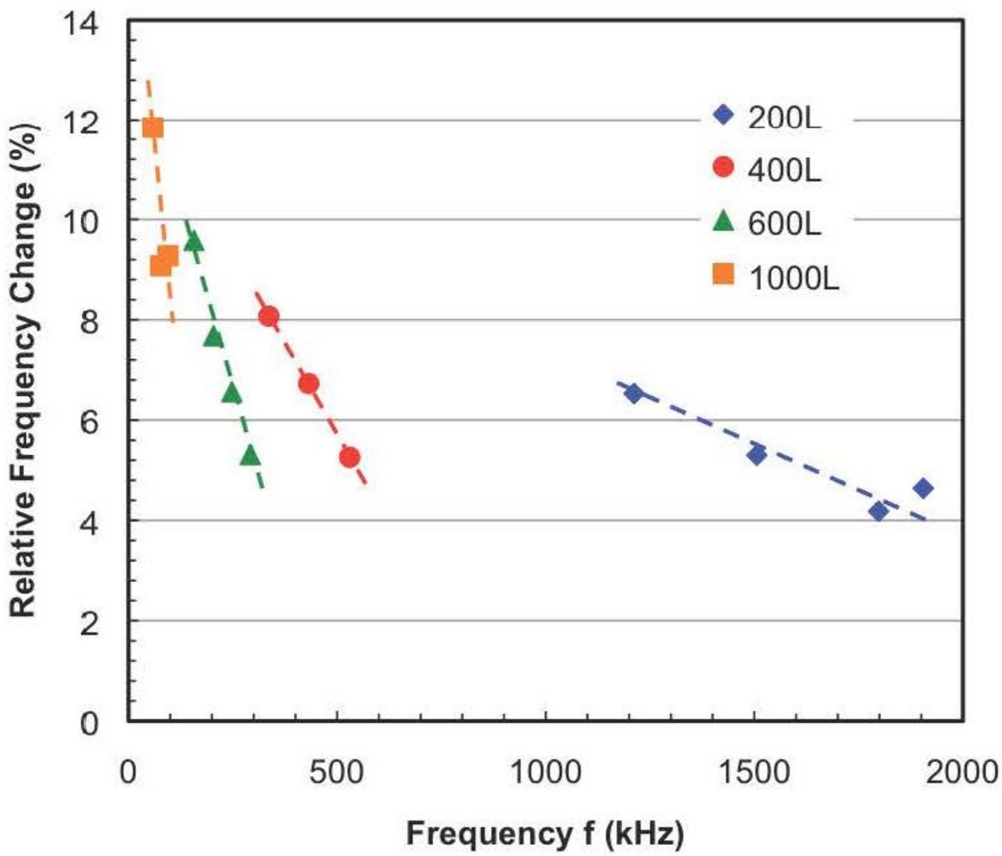

Figure 4. Relative frequency change associated with immersing the cantilevers from air into water as a function of the in-plane resonance frequency in air. Note that for a given length, the resonance frequency increases with increasing cantilever width

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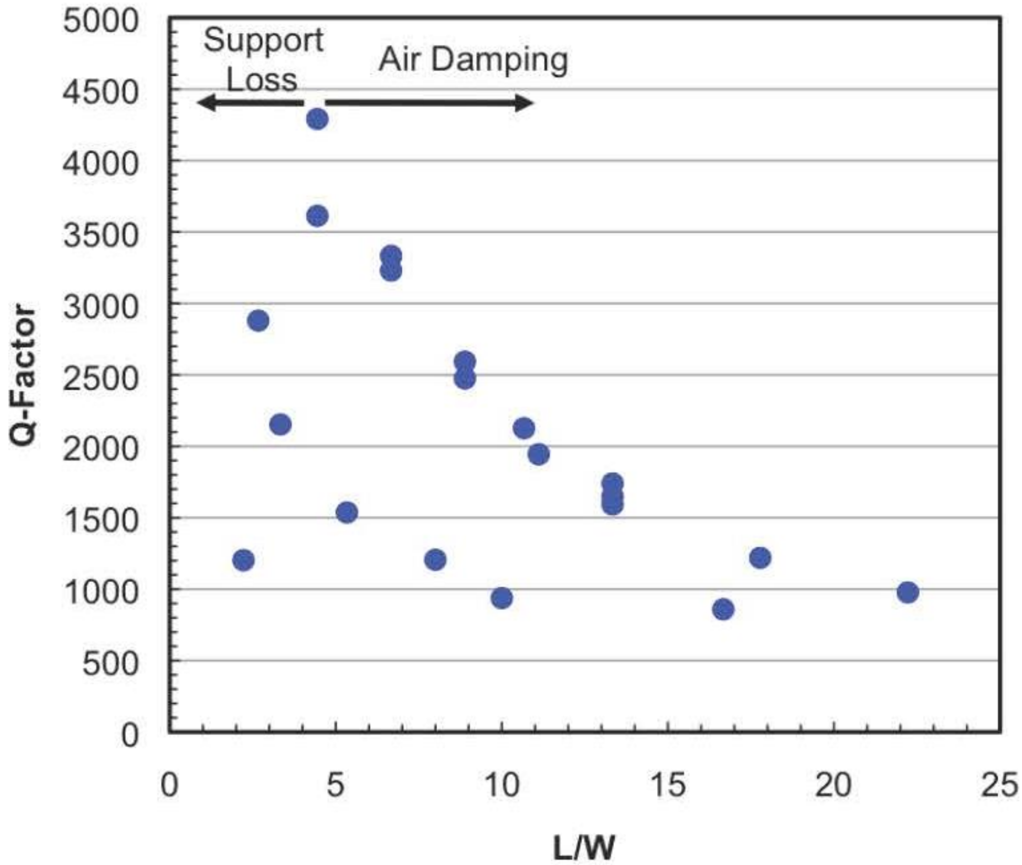

Figure 5. Q-factor of fundamental in-plane flexural mode for cantilevers with 12μm silicon thickness in air as a function of the length-to-width ratio

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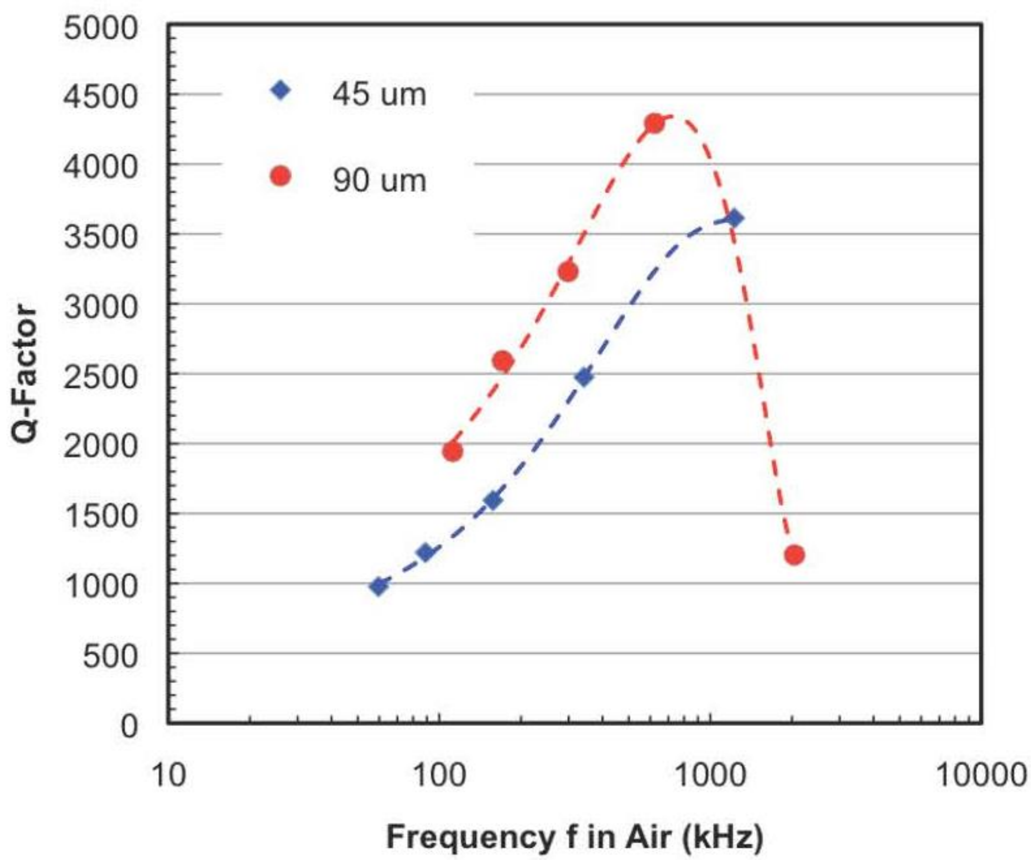

Figure 6. Q-factor of in-plane bending mode for 45μm and 90μm wide cantilevers with 12μm silicon thickness in air as a function of the resonance frequency

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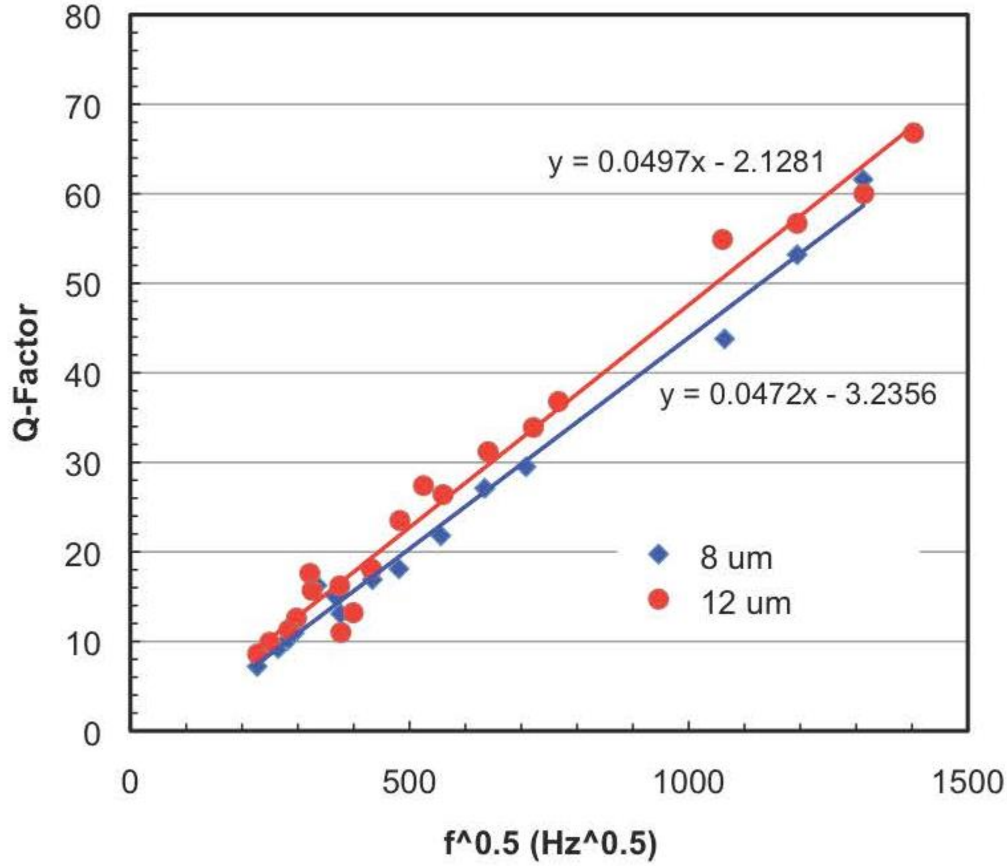

Figure 7. Q-factor of in-plane bending mode for cantilevers with 8μm and 12μm silicon thickness in water as a function of the square-root of the resonance frequency

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