



Detection of the Au thin-layer in the Hz per picogram regime based on the microcantilevers

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Abstract

Through the use of oscillating microcantilevers, a micromechanical mass detection with a resolution of a Hz per picogram regime is reported. Through MEMS processes, piezoelectric microcantilevers that are simultaneously capable of self-actuation and the electrical measurement of resonant frequencies were fabricated. Mass detection in the Hz per picogram regime is demonstrated with a deposition of an Au thin-layer, of which the thickness is precisely controlled. In addition, it is shown that a scaling down of the microcantilevers enhances the sensitivity during the micromechanical mass detection.

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1. Introduction

Microcantilevers have played an important role in both MEMS and bio-MEMS. In particular, recent studies suggest that microcantilevers have a great potential as biosensors for the label-free detection of biomolecules related to specific diseases [1,2]. The microcantilevers allow a label-free detection of biomolecules such as prostate specific antigen (PSA) or myoglobin by measuring the deflection change and/or resonant frequency shift driven by biomolecular interactions on the microcantilever surface [1,3,4]. Furthermore, the microcantilever can be used as a nanomechanical sensor for detecting nanoparticles [5,6] and/or a molecular layer [7–10] by measuring the deflection change and/or resonant frequency shift induced by the adsorption of the molecular layer on the microcantilever surface.

The principle for a label-free detection with microcantilevers is that the molecular adsorption on the microcantilever surface generates a flexural rigidity change and also affects the mass of a microcantilever so as to alter the deflection and/or the resonant frequency. Static-mode microcantilevers are capable of a label-free detection by measuring static deflection, while oscillating microcantilevers utilize the resonant frequency shift for a label-free detection. For static-mode microcantilevers [1,3,11], an optical apparatus is required to measure the deflection, causing difficulty in miniaturization. Further, if the deflection due to molecular adsorption is exceedingly small, difficulties may arise while measuring the deflection change at a low biomolecule concentration with the optical apparatus. For conventional oscillating microcantilevers, required is an external actuator, thus miniaturization is not likely possible. However, a recent work by the authors investigated oscillating piezoelectric microcantilevers that did not require an external actuator or optical apparatus. Consequently, the microcantilevers developed in that study may be more appropriate for miniaturized sensor applications [12–14]. In particular, these microcantilevers employ piezoelectric and converse piezoelectric effects, enabling self-actuation and an electric measurement of the resonant frequencies.

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Recently, several cases of biomolecule detections using a piezoelectric cantilever have been reported. Biomolecule detection was accomplished by monitoring the resonant frequency shift of a piezoelectric cantilever over time. In a biomolecular interaction, the resonant frequency shift was mainly affected by the induced surface stress due to the electrostatic interaction between biomolecules. Herein, the mass contribution to the resonant frequency shift was minimized, as the mass of the reacted biomolecule monolayer on the cantilever surface was negligible. Another possible application of the piezoelectric cantilever is as a mass sensor that is similar to quartz crystal microbalance (QCM), for instance, a cantilever sensor to monitor thin film deposition. Lee et al. [15] reported that a resonant frequency shift during the deposition of an Au thin film was mainly affected by the Au mass. Accordingly, a piezoelectric cantilever is seemingly viable as a mass sensor to monitor thin film deposition in a wide thickness range.

In this article, a mass detection with a sensitivity rating of an order of 1 Hz/pg is reported using piezoelectric microcantilevers. Very small piezoelectric cantilevers with different sizes were fabricated through a MEMS processes. Through a deposition of an Au thin-layer as a mass source, the mass sensitivity of each cantilever was measured. From the experimental results, the effect of the cantilever length in 10 μm orders on the mass sensitivity is discussed.

2. Theory

The dynamic response of a microcantilever is well characterized by the following equation of motion.

$$\xi \frac{\partial^4 w(x, t)}{\partial x^4} + \mu \frac{\partial^2 w(x, t)}{\partial t^2} = 0 \quad (1)$$

Here, $w(x, t)$ is the deflection, ξ the effective bending modulus and μ is the effective mass per unit length for a microcantilever. It is noted that the effective mass per unit length, μ , can be easily estimated by the rule of mixture [16,17], whereas the elasticity theory [18] enables the evaluation of the effective bending modulus ξ for a microcantilever consisting of multiple layers (Fig. 1, Table 1), such as Ta/Pt/PZT/Pt/SiO₂ for self-actuation, and electrical sensing such as $\xi = \sum E_k I_k$, where N is the total number of layers (i.e. $N=5$), E_k Young's modulus for the k -th layer, and I_k is the moment of inertia for the k -th layer. An analytical solution for Eq. (1) provides the resonant frequencies f_i of the microcantilevers.

$$f_i = \frac{1}{2\pi} \left(\frac{\lambda_i}{l} \right)^2 \sqrt{\frac{\xi}{\mu}} \quad (2)$$

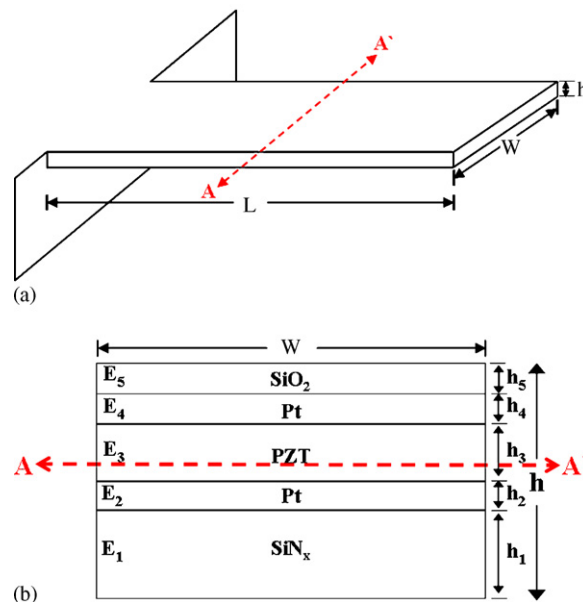


Fig. 1. Schematics of (a) a PZT microcantilever and (b) a cross-sectional view of the PZT microcantilever. The line AA' represents the neutral axis.

where l is the cantilever length, and λ_i represents eigenvalues satisfying the transcendental equation of $\cos \lambda_i \cosh \lambda_i + 1 = 0$.

In a case in which a molecular mono-layer is adsorbed on the microcantilever surface, it is assumed that molecular adsorption onto the microcantilever surface does not induce intermolecular interactions that alter the dynamic response of the microcantilevers, and that the molecular adsorption does not change the flexural rigidity of the microcantilever. The resonant frequency after the molecular adsorption is

$$\bar{f}_i = \frac{1}{2\pi} \left(\frac{\lambda_i}{l} \right)^2 \sqrt{\frac{\xi}{\mu + \Delta\mu}} \quad (3)$$

where $\Delta\mu$ is the added mass per unit length for a molecular layer (i.e. the total mass of the molecular layer is $\Delta m = l\Delta\mu$). From Eqs. (2) and (3), the induced mass Δm of a molecular layer is

$$\Delta m = \frac{\lambda_i^4}{4\pi l^3} \xi \left(\frac{1}{\bar{f}_i^2} - \frac{1}{f_i^2} \right) \quad (4)$$

Eq. (4) allows the evaluation of the mass of the molecular layer adsorbed onto the surface by measuring the resonant frequency before and after the molecular adsorption on the surface.

For the adsorption of a minute molecular layer on a surface, i.e. $\Delta\mu \ll \mu$, the resonant frequency shift due to the adsorption

Table 1
Parameters of the PZT microcantilever layers composed of SiN_x/Pt/PZT/Pt/SiO₂

Material	Thickness, $h(\times 10^{-9})$ [m]	Thickness ratio	Young's modulus, $E(\times 10^{10})$ [N/m ²]	Density, ρ [kg/m ³]
SiO ₂	200	0.16	7.30	2500
PZT	500	0.40	7.70	21400
Pt	250	0.20	16.80	7500
SiN _x	700	0.24	29.00	3100

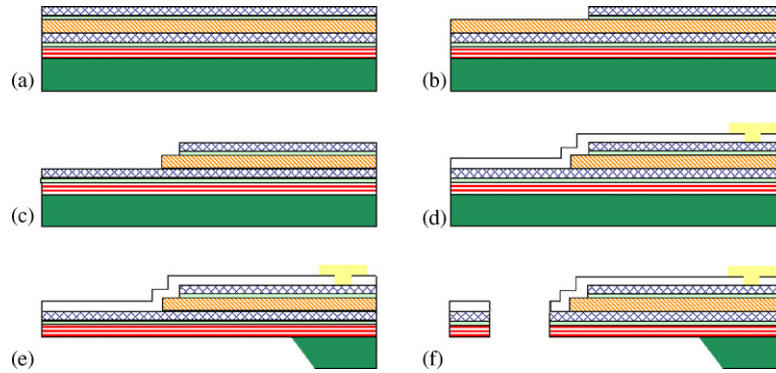


Fig. 2. Fabrication flow chart and structure of the nano-mechanical PZT cantilever for self-actuation/sensing. (a) thin film deposition SiN_x/Si/SiN_x/Ta/Pt/PZT/Pt; (b) Ta/Pt etching using ICP; (c) PZT etching using ICP; (d) SiO₂ deposition, via hole etching and Au lift-off; (e) Si etching using KOH etchant; (f) SiN_x/Pt/SiO₂ etching for releasing window using RIE/ion milling.

on the surface is approximated as follows:

$$\Delta f_i \equiv \bar{f}_i - f_i = -\frac{1}{2} f_i \frac{\Delta \mu}{\mu} = -\frac{1}{2} f_i \frac{\Delta m}{m} \quad (5)$$

The resonant frequency shift with respect to the loaded mass is

$$\frac{\Delta f_i}{\Delta m} = -\frac{1}{2} \frac{f_i}{m} = -\frac{\lambda_i^2}{4\pi} \frac{1}{bl^3} \frac{1}{\rho} \sqrt{\frac{\bar{E}}{\rho}} \quad (6)$$

Here, ρ is the effective density, i.e. $\rho = \mu/bh$, where b is the width and h is the thickness. \bar{E} is the effective Young's modulus defined as $\bar{E} = \xi/bh^3$. Eq. (6) provides that the mass sensitivity $\Delta f_i/\Delta m$ is inversely proportional to bl^3 .

3. Experimental procedure

3.1. Fabrication of the self-actuating microcantilevers

We fabricated the piezoelectric microcantilevers, consisting of multi-layers of Ta/Pt/PZT/Pt/SiO₂ on a SiN_x supporting layer, for electrical self-actuating and sensing without any external oscillators or an optical apparatus. Fig. 2 shows the fabrication process of the PZT microcantilever. For the piezoelectric capacitor, p-doped Si (1 0 0) wafer (525 ± 20 μm thickness) substrates with a 100 mm-diameter were utilized for the deposition of low-stress 0.7 μm silicon nitride (SiN_x) layers using low-pressure chemical vapor deposition (LPCVD). A Ta layer with a thickness of 0.03 μm allowed a bottom electrode layer (Pt layer) with a thickness of 0.15 μm to be bound to the SiN_x layer. The PZT films were deposited on the bottom electrode by the spin coating of a mixed PZT solution (diol-based sol–gel route) at 3000 rpm for 30 s. They were subsequently fired at 400 °C for 5 min and then annealed at 650 °C for 10 min. For a metal-ferroelectric-metal (MFM) capacitor structure, a Pt layer was deposited onto a PZT film as a top electrode via RF sputtering. The Pt layer as a top electrode in addition to the PZT layer was etched by inductive coupled plasma (ICP) etching. A SiO₂ thin film with a thickness of 0.2 μm was deposited onto the top electrode via a plasma-enhanced chemical vapor deposition (PECVD) process. Following this, contact holes on the top

electrode were etched. The contact electrode and the top electrode were connected through an Au lift-off process. The pattern of the Pt bottom electrode was conducted with ICP, while the pattern of the bottom SiN_x window was done by reactive ion etching (RIE). The bulk silicon (p-doped Si wafer) was then etched using KOH. Finally, the etching of the SiN_x layer (top SiN_x layer on Si wafer) was implemented via RIE. After the fabrication of the piezoelectric microcantilever, a SiO₂ thin film with thickness of 1000 Å was deposited by means of PECVD as an electrical, chemical passivation layer. Fig. 3 shows an SEM image of piezoelectric microcantilevers with the varying dimen-

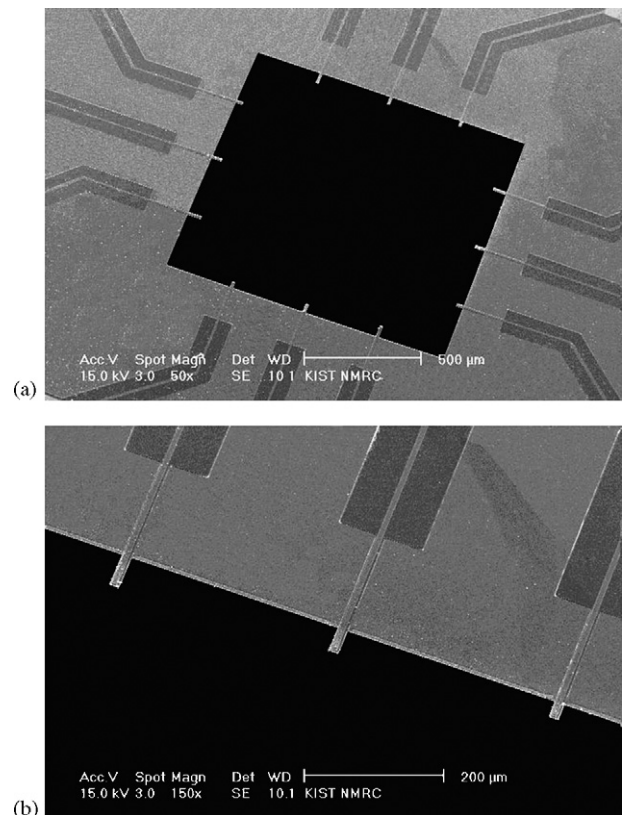


Fig. 3. SEM images of the PZT microcantilever. (a) An array chip and (b) individual cantilever devices with dimension of 15 μm × 35 μm, 15 μm × 50 μm and 15 μm × 60 μm ($W \times L$) cantilever, respectively.

sions of $15\ \mu\text{m} \times 60\ \mu\text{m}$, $15\ \mu\text{m} \times 50\ \mu\text{m}$, and $15\ \mu\text{m} \times 35\ \mu\text{m}$ (width \times length).

3.2. Measuring the resonant frequency shift due to induced mass of the gold layer

In order to measure the resonant frequency of the microcantilevers, we used an impedance analyzer (4292A, Agilent technologies, USA), which enabled the monitoring of the phase angle as a function of the sweeping frequency. Specifically, while the phase angle of the impedance for a microcantilever that functions as a capacitor is approximately -90° in the off-resonance, the phase angle in the resonant frequency exhibits the peak value that is responsible for the microcantilever motion.

The resonant frequency shift induced by the mass of the Au was measured using an impedance analyzer. The resonant frequencies of the microcantilever were measured before and after the deposition of the Au layer, and the resonant frequency shift was then calculated. The Au thin-layer was deposited on the microcantilever with thicknesses of 6, 12, 32, and $50\ \text{\AA}$ using the thermal evaporator. The thickness of the Au layer was precisely controlled by a thickness/rate monitor system (Sycon Instruments, USA), which was a component of the thermal evaporator. From these measured resonant frequencies, it was possible to estimate the resonant frequency shift induced by the Au layer.

4. Result

4.1. Dynamic behavior of the microcantilevers based on the length scale

As stated earlier, the impedance analyzer enables the measurement of the resonant frequency of microcantilevers. Fig. 4 shows the phase angle change as a function of the sweeping frequency of the proposed fabricated microcantilever with a dimension of $20\ \mu\text{m} \times 80\ \mu\text{m}$. At off-resonance, the phase angle is not quite -90° due to parasitic impedance generated by the cable and the probe connecting the electrode of the cantilever to the impedance analyzer. However, the phase angle shift in

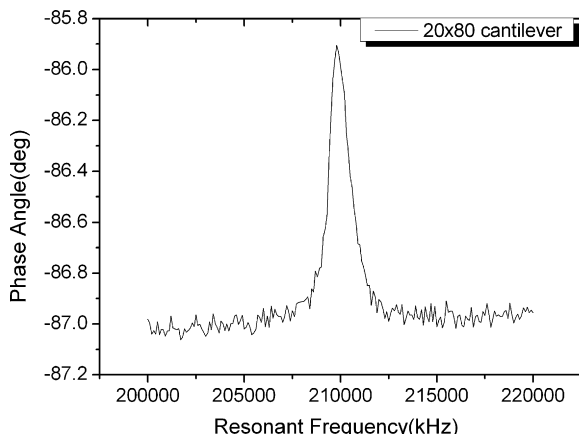


Fig. 4. Phase angle with respect to the first resonant frequency of the $20\ \mu\text{m} \times 80\ \mu\text{m}$ ($W \times L$) cantilever.

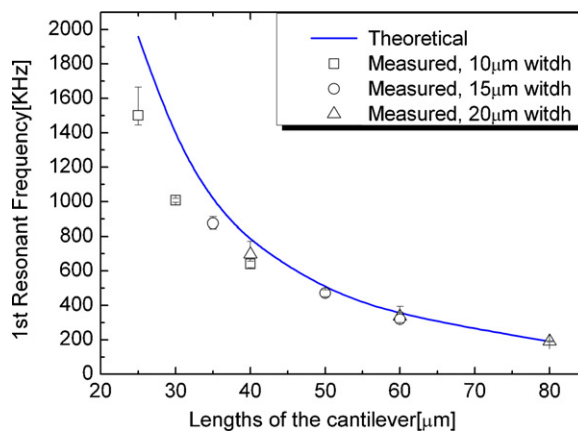


Fig. 5. Theoretical and measured first resonant frequencies of the PZT cantilever vs. the cantilever length (L). The solidus line represents the theoretical results, and (\square), (\circ) and (\triangle) represent the measured first resonant frequencies of the 20, 15 and 10 μm width cantilever, respectively.

the resonance was much larger than phase angle shift originated from the parasitic impedance, thus the parasitic impedance is negligible in the measurement of the resonance frequency.

In Fig. 5, it is shown that the size effect plays a role on the dynamic response of microcantilevers. Specifically, the length scale plays a vital role on the dynamic response, whereas the width scale does not affect the dynamic response. This is consistent with the theoretical model in which the resonant frequency is dependent only on the length. That is, the resonant frequency is inversely proportional to the square of the length, as suggested in Eq. (2). The experimental data for resonant frequencies of microcantilevers with a length scale larger than $50\ \mu\text{m}$ are quantitatively identical to the theoretical predictions, whereas for microcantilevers with a length scale less of than $50\ \mu\text{m}$, the theoretical model does not allow a precise prediction of the resonant frequencies (see Fig. 5). Furthermore, the deviation of the experimental data for the resonant frequency from the theoretical model becomes larger as the length scale of the microcantilevers becomes smaller. The difference between the experimental data and the analytical predictions can be ascribed to the etching fabrication process, which can affect the SiN_x supporting layer. That is, for a small length scale that increases the resonant frequency enormously, the effect of the etching fabrication that changes the thickness and flexural rigidity may induce a significant deviation of the resonant frequency obtained by experiments from the analytical expectations. Moreover, deviation of the measured resonant frequency from theoretical predictions can be attributed to the photolithography fabrication process. During this process, corners of the mask-defined patterns are exposed to undesired scattered light whereas several areas outside the mask-defined pattern, especially the anchors of the cantilever, are exposed to a smaller amount of light compared to the other non-masked area. These circumstances result from the diffraction/scattering of the incident UV-light near the corners or anchors of the pattern. Accordingly, the photoresist around the corners and anchors are not perfectly developed, leading to a reduction of pattern fidelity [19]. As the cantilever dimension becomes smaller, this effect leads to a more severe alteration of the dimension of the

Table 2
Loaded Au mass on each cantilever surface with three different dimensions

Au thickness (Å)	Loaded mass (pg)		
	20 μm × 80 μm	15 μm × 60 μm	10 μm × 30 μm
6	18.58	10.42	3.47
18	55.58	31.27	10.42
50	154.4	86.85	28.95
100	308.8	173.7	57.9

cantilever, further resulting in a deviation from the expected resonant frequency.

4.2. Sensitivity for detecting the mass of Au layer on the microcantilever

For the study of the role of the size effect on the sensitivity of microcantilevers for the mass detection of an Au layer, the dynamic response change of microcantilevers is considered, using several sizes (20 μm × 80 μm, 15 μm × 60 μm and 10 μm × 30 μm), upon which an Au thin film layer with several thicknesses (6, 12, 32, and 50 Å) is deposited. The effective loaded mass of the Au thin film layer for each thickness on each microcantilever is provided in Table 2. Here, the effective loaded mass is simply calculated from the density and volume of the Au thin film layer. Fig. 6 shows the resonant frequency shift as a function of the loaded mass for microcantilevers with different sizes. In Fig. 6, it is observable that size plays a role on the mass sensitivity such that the microcantilever with a dimension of 10 μm × 30 μm exhibits a much better mass sensitivity compared to the two other microcantilevers with larger lengths and width scales. Furthermore, the mass sensitivity $\Delta f_i/\Delta m$ from the experiments is quantitatively similar to the theoretical predictions, with the exception of the microcantilever with a dimension of 10 μm × 30 μm. For the microcantilever with a dimension of 10 μm × 30 μm, the deviation of the mass sensitivity obtained by the experiments from the theoretical prediction may be

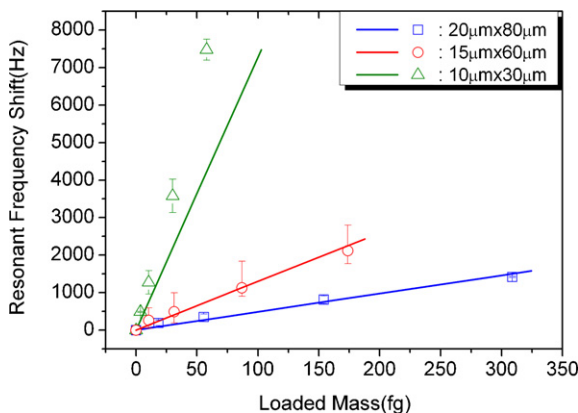


Fig. 6. Theoretical and measured first resonant frequency shift of the PZT cantilever vs. loaded Au mass (Δm) on the cantilever. The solidus line indicates the theoretical results and (□), (○) and (△) indicate the resonant frequency shifts of the 20 μm × 80 μm, 15 μm × 60 μm and 10 μm × 30 μm ($W \times L$) cantilever due to the Δm .

ascribed to the etch fabrication process, as stated earlier. Specifically, the mass sensitivities are 4.88, 13.16, and 128.37 Hz/pg for microcantilevers with dimensions of 20 μm × 80 μm, 15 μm × 60 μm, and 10 μm × 30 μm, respectively. For the microcantilever with a dimension of 10 μm × 30 μm, the theoretical prediction, using the experimental data of the resonant frequency shift from Eq. (6), on the mass sensitivity is 76.92 Hz/pg.

As suggested in the theory, the mass sensitivity $\Delta f_i/\Delta m$ is related to the width and the length such as $\Delta f_i/\Delta m \sim 1/bl^3$. Here, the mass sensitivity $\Delta f_i/\Delta m$ is denoted as α , i.e. $\alpha = \Delta f_i/\Delta m$. Two microcantilevers are considered, for example, Cantilever (1) and Cantilever (2). From Eq. (6), the ratio of the mass sensitivity between the two microcantilevers is given as

$$\frac{\alpha^{(2)}}{\alpha^{(1)}} = \left[\frac{b^{(1)}}{b^{(2)}} \right] \left[\frac{l^{(1)}}{l^{(2)}} \right]^3 \quad (9)$$

Here, the superscript (i) indicates the microcantilever (i), where $i = 1, 2$. Taken into account are the two microcantilevers whose dimensions are 10 μm × 30 μm [Cantilever (1)] and 15 μm × 60 μm [Cantilever (2)]. From the experiments, the ratio of the mass sensitivity is $\alpha^{(2)}/\alpha^{(1)} = 0.1025$, while the theoretical prediction from Eq. (9) results in the ratio of the mass sensitivity of $\alpha^{(2)}/\alpha^{(1)} = 0.08333$. This indicates that the mass sensitivity obeys the rule that $\Delta f_i/\Delta m$ is inversely proportional to bl^3 .

5. Conclusion

In this article, microcantilevers that can detect the induced mass of an Au thin film layer in the order of 1 Hz/pg are reported. This study shows that the scaling down of the microcantilever significantly increases the mass sensitivity. Specifically, it is shown that the mass sensitivity is related to the width and the length, given as Eq. (6). This relationship suggests that small length-scale microcantilevers are appropriate for the monitoring of depositions of thin films with a high mass sensitivity. That is, microcantilevers operated in vibration (oscillation) mode enable the possibility of miniaturization, while simultaneously enhancing mass sensitivity. Thus, these self-actuating microcantilevers exhibit a high potential for utilization as sensors and/or biosensors.

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Dong Won Chun was born in 1978 in Seoul, South-Korea. He received the BS degree in Department of Materials Science and Engineering from the Yonsei University, Seoul, where he is currently working toward the MS degree in Department of Materials Science and Engineering. He is interested in MEMS, nanomechanics, MEMS sensor and integrated micro/nano device.

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Jeong hoon Lee is a Postdoctoral Associate at Massachusetts Institute of Technology (MIT), USA. He is currently a member of the Micro/Nanofluidics BioMEMS Group at Research Laboratory of Electronics (RLE). His main research in MIT is the development of nanofluidic systems and Macro-to-micro/nano interfaces. He received the BS and MS degrees in ceramic engineering from the Yonsei University, Seoul, Korea, in 1997, and 1999. He received his PhD from Yonsei University in 2004. He specialized in MEMS/Nanomechanics. From 1999 to 2005, he was a research scientist in the Microsystem Research Center at Korea Institute of Science and Technology (KIST) in Seoul, Korea. He is interested in the application and development of MEMS, nanomechanics, integrated micro/nano devices and self assembled monolayer (SAMs) formation.

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