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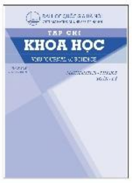

Original Article

Impact of Geometry Dimension on Quality Factor and Mass-Sensitivity of PZT Thin Film-based Microcantilevers

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Abstract: The impact of the piezoelectric cantilever geometry and resonant mode on the quality factor and mass-sensitivity was investigated. The piezoelectric cantilevers with $Pb(Zr_{0.52}Ti_{0.48})O_3$ (PZT) as an active layer were fabricated based on MEMS technology. The PZT thin films were grown on the silicon-on-insulator substrates by pulsed laser deposition. It is shown that the resonant frequency of a defined vibration mode is inversely proportional to the length squared of the piezoelectric cantilever. The experimental results indicate that a shorter cantilever length contributes to a larger quality factor. The cantilever quality factor has values in the range of 190 to 505, depending on the length and resonant mode. The mass-sensitivity increases when the cantilever length decreases and reaches the highest value of 37.6 Hz/pg for the 100-µm-long cantilever. High-mode vibration was successfully exhibited for the higher mass-detection sensitivity.

Keywords: Piezoelectric cantilever, MEMS, PZT thin film, mass-sensitivity

1. Introduction

In recent years, with MEMS fabrication and piezoelectric film deposition technologies progressing rapidly, micromachined piezoelectric resonant mass sensing devices have emerged as highly versatile sensors for detecting gases, chemicals, and biological entities [1–8].

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The piezoelectric cantilevers, in comparison to conventional ones, have advantages such as not requiring an external actuator or optical apparatus. In particular, these cantilevers employ piezoelectric effects, enabling self-actuation and an electric measurement of the resonant frequencies. Being viable as mass sensor, the current research trend has focused on the demonstration of the sensing capability of piezoelectric cantilevers. Ferrari *et al*. [9] have reported on the mass-sensitivity of 1-mm-long alumina beam with PZT thin film for piezoelectric actuation and detection. The mass-sensitivity is measured by depositing a known quantity of silicone grease on the cantilever and found to be equal to 0.9 µg/Hz. Shin *et al*. [10] have shown better mass-sensitivity with 200-µm-long cantilevers including 500 nm thick PZT thin films with resonant frequencies ranging from 18.6 to 24.2 kHz. The mass-sensitivity has been measured by depositing thin copper films on cantilever and found to be in the 30 pg/Hz. Similar results with 380, 480 and 580-µm-long and 400-µm-wide cantilevers containing 2 µm thick PZT films have reported by Park *et al*. [11]. The mass-sensitivity has been measured there by depositing a thin gold film on the cantilever tip and found to be 152, 57.1 and 30.7 pg/Hz, respectively. In a recent study, Nguyen *et al*. [12] have shown that the mass-sensitivity of 12.45 pg/Hz was obtained by optimizing the thickness and composition of PZT thin film for the piezoelectric cantilevers. To improve the mass-sensitivity of the piezoelectric cantilever, however, it is shown that few researches have performed the geometric optimization as well as studies on the effect of geometric dimension on the properties of the piezoelectric cantilevers.

In this study, the piezoelectric cantilevers with the dimension of 100-µm in width and 100–800-µm in length and the $Pb(Zr_{0.52}Ti_{0.48})O_3$ (PZT) thin film as an active layer were fabricated based on MEMS technology. The dependence of quality factor and mass-sensitivity on length and vibration resonant mode of piezoelectric cantilevers has been investigated.

2. Experimental procedure

2.1. Thin Film and Piezoelectric Cantilever Fabrication

In the PZT thin films fabrication process, the textured films were grown on $Pt/Ti/SiO_2/SOI$ substrate. The $Pb(Zr_{0.52}Ti_{0.48})O_3$ thin films were deposited using pulsed laser deposition (PLD) method with a KrF excimer laser source (Lambda Physik, 248 nm wavelength) performed at 3.5 J/cm^2 laser-fluency and 10 Hz repetition rate. The target-substrate distance was kept at 6.0 cm. The PZT thin films were grown at substrate temperature of 600 °C and ambient oxygen pressure of 0.1 mbar. After deposition, the films were cooled down to room temperature in oxygen atmosphere with a ramp rate of 6 °C/min.

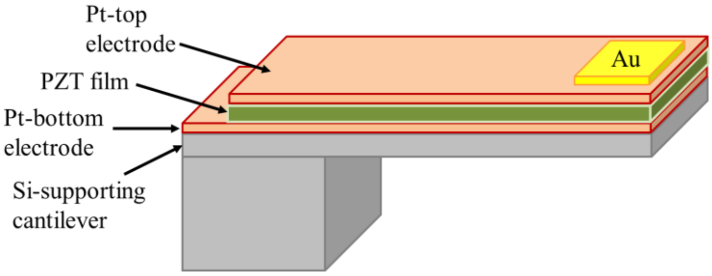

Figure 1. Schematic structure representation of PZT-based piezoelectric cantilever.

Figure 1 represents the structure of PZT-based piezoelectric cantilever. The piezoelectric cantilever consists of a Si supporting beam, which is covered by a 1-µm-thick-PZT layer having 100-nm-thick Pt bottom and top electrodes. In this study, the Si supporting beams have 100-µm in width, 100–800-µm in length and 10-µm thick. A dimension of 60×60 µm^2 gold sensing layer is located on the tip of the piezoelectric cantilever. The fabrication process of the piezoelectric cantilevers using PZT thin films is shown in Figure 2. Devices were fabricated on a p-type (001) silicon-on-insulator (SOI) substrate (size: 20×20 mm^2) with a 10±0.5 µm-thick device layer, a 2±0.1 µm-thick buried-oxide (BOX) layer and a 380±20 µm-thick handle layer (Figure 2a).

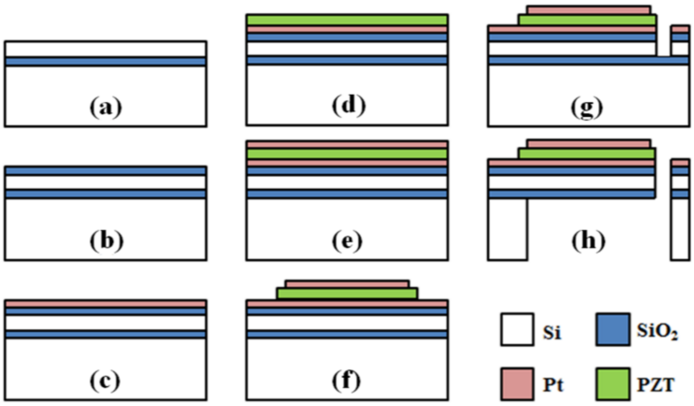

Figure 2. Fabrication process of the piezoelectric PZT cantilever using SOI substrates.

The SOI wafer was cleaned by a standard cleaning process and oxidized by wet thermal oxidation at 1100 °C for 1 hour to form a 500 nm thick SiO_2 layer (Figure 2b). Then, the Pt(100-nm)/Ti(15-nm) bottom electrode was deposited by sputtering (Figure 2c). In the following step, a 1-µm PZT layer was deposited by pulsed laser deposition (Figure 2d). After that, the Pt(100-nm)/Ti(15-nm) top electrode was deposited by sputtering on the surface of PZT layer (Figure 2e). The fabrication of piezoelectric cantilevers was conducted by the MEMS microfabrication process, including photolithography and etching. First, the top Pt/Ti electrode was etched by Ar-ion beam etching and the PZT film was removed by sequential wet-etching with HF and HCl solutions (Figure 2f). Following this step, the bottom Pt/Ti electrode was also Ar-ion beam etched and the Si device layer was etched by a deep reactive-ion etching (DRIE) using a mixture of SF_6, O_2 and C_4F_8 gases (Figure 2g). Finally, the release etch for forming cantilevers was performed by the DRIE (Figure 2h)

2.2. Characterization

The crystalline structures of the PZT thin films were analyzed by X-ray diffraction (XRD: Philips X'PERT MPD). The X-ray source is a long-fine-focus, ceramic X-ray tube with Cu anode (wavelength: 1.5405 Å). Normal operating power is 1.8 kW (45 kV and 40 mA).

The polarization hysteresis (P-E) loop measurements were performed based on the ferroelectric mode of the aixACCT TF-2000 Analyzer using a triangular ac-electric field of ±200 kV/cm at 1 kHz frequency and at room temperature.

To evaluate the performance of cantilever beams as a mass-sensing device, oscillation characteristics were measured as a function of frequency using a Polytech MSA-400 scanning laser Doppler vibrometer (LDV) method. In this measurement, the *ac* sine-wave of 1 V_{p-p} (peak-to-peak) superimposed with a *dc* voltage of 2 V in the frequency range from 0 to 2000 kHz was applied to the actuation top-electrode while the bottom-electrode was grounded. A shift of the resonance frequency is induced by the mass of the substance added to the gold (Au) sensing layer.

3. Results and Discussion

Figure 3a shows x-ray diffraction (XRD) θ-2θ scans of the PZT thin films grown on Pt/Ti/SiO_2/SOI substrates by the PLD method. A (100)-preferred orientation in the PZT films is observed. It is shown that no evidence of pyrochlore phase formation is observed within the resolution limits of the XRD. The PZT film also reveals a small admixture of (110) and (111)-orientation. The room-temperature polarization hysteresis loop of PZT film is shown in the Figure 3b. The remnant polarization (P_r) and the coercive field have values of 16.8 µC/cm^2 and 28.5 kV/cm, respectively. These values are consistent with the reported ones [13], which indicates the good quality of PZT film.

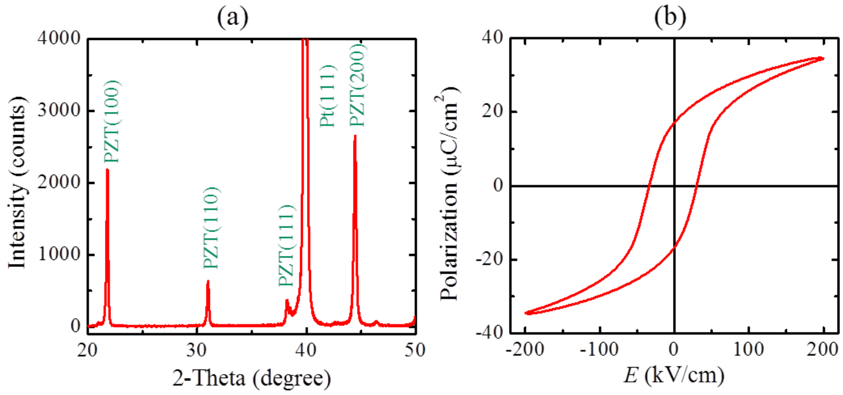

Figure 3. (a) X-ray diffraction (XRD) pattern and (b) Polarization hysteresis (P-E) loop of 1-µm-thick PZT thin film grown on Pt/Ti/SiO_2/SOI substrate.

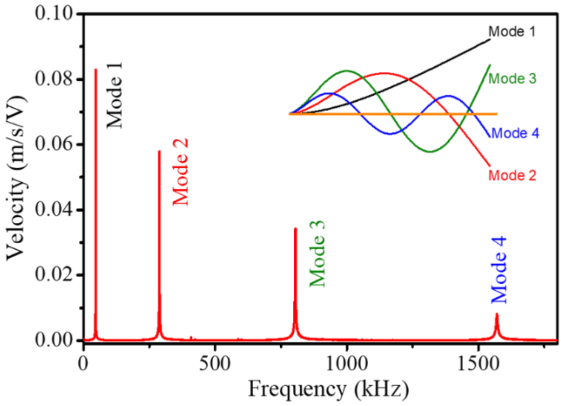

Figure 4. Vibration resonant spectrum of 500-µm-long piezoelectric cantilever, measured using LDV method in the range of 0–2000 kHz. The *inset* shows the vibration waveforms for the first four bending modes of cantilever.

Figure 4 shows peaks of the first four resonant modes experimentally obtained on the piezoelectric cantilever having dimensions of 500-µm in length, 100-µm in width and 11-µm total device thickness (including 1-µm thickness of PZT layer and 10-µm Si supporting layer). Figure 4 *inset* represents the vibration waveforms for the first four bending modes of the cantilever. The resonant frequencies of these modes have values of 46.06 kHz (1st mode), 288.6 kHz (2nd mode), 804.9 kHz (3rd mode), and 1570.8 (4th mode), respectively.

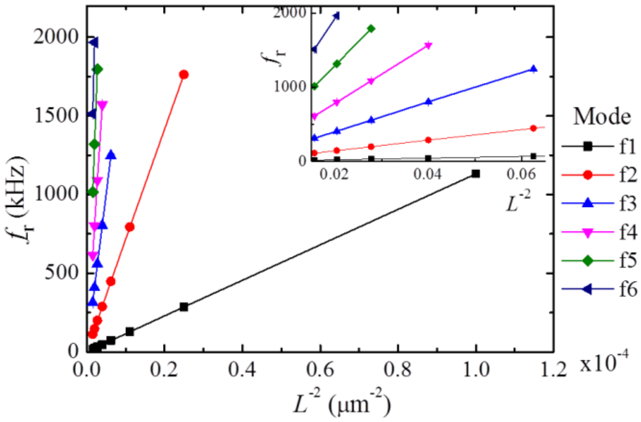

Figure 5. Measured mode resonant frequency (f_r) versus inverse of the square length (L^{-2}) of the cantilevers. The *inset* presents the zoomed-in plot.

The resonant frequency as the function of the length for the piezoelectric cantilevers with 100-µm in width and varying lengths (100–800 µm) is shown in Figure 5. The obtained results indicate that the resonant frequency of a defined mode is inversely proportional to the length squared of the piezoelectric cantilever.

For a piezoelectric cantilever of length L, the resonant frequency can be given by [14]:

$$f_n = \frac{\lambda_n^2}{2\pi}\frac{t}{L^2}\sqrt{\frac{E}{12\rho}} \tag{1}$$

where E is the elastic modulus, ρ the density, t the thickness, and L the length of the piezoelectric cantilever. The eigenvalue λ_n depending on the shape of n^{th} resonant mode has values of 1.875, 4.694, 10.995, 14.14, and 17.28 for 1st mode ($n = 1$), 2nd mode ($n = 2$), 3rd mode ($n = 3$), 4th mode ($n = 4$), and 5th mode ($n = 5$), respectively. It is shown that the experimental results of the resonant frequency as function of the piezoelectric cantilever length are in good agreement with the equation (1).

In various applications, piezoelectric cantilever-based mass sensors would have a high value of mass-sensitivity, which depends on quality factor (Q-factor). This parameter is extensively used in characterizing the mechanical response of the cantilevers. The Q-factor can be defined from the resonant curve as [15, 16]:

$$Q_n = 2\pi \frac{E_{max}}{E_{\mathrm{lost}}\frac{f_n}{\Delta f_n}} \tag{2}$$

where E_{max} is the maximum energy stored, E_{lost} is the total energy lost in a period; f_n and Δf_n *are* the resonant frequency and the full-width at half-maximum (FWHM) of the n^{th} resonant mode, respectively.

Figure 6 illustrates the Q-factors of the cantilevers as a function of the resonant mode for varying length (100–800) µm. The Q-factor examined at the fundamental resonant modes increases from 267 to 678 when the length decreases from 800 to 100-µm. It is shown that a shorter cantilever length will contribute to a larger Q-factor at the fundamental resonant modes, which can be explained by the air damping effect [17].

The influence of resonant mode on the Q-factor depends on the cantilever geometry dimensions. In the measured frequency range from 0 to 2000 kHz, the Q-factor decreases with the higher-order resonant modes for the cantilevers with the length of 200–300 µm. Meanwhile, for the cantilevers with the length

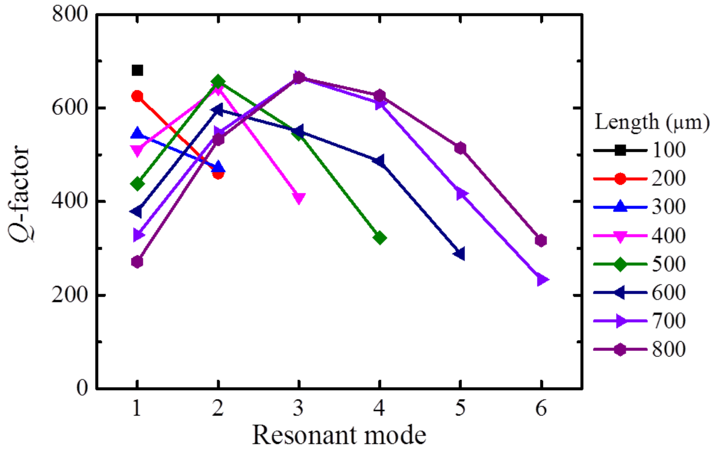

Figure 6. Measured quality *Q*-factor dependence on resonant mode and cantilever length.

of 400–600 µm, the *Q*-factor firstly increases and reaches the maximum value at the 2nd resonant mode, and then decreases with the higher-order resonant modes. The *Q*-factor exhibits the same behavior for the cantilevers with the length of 700–800 µm, however, the maximum value of the *Q*-factor is obtained at the 3rd resonant mode. The increase of *Q*-factor with the increase of resonant mode for cantilevers with the length of 400–800 µm can be due to the increase of the vibration frequency [17]. In the case of the high-order modes, the decrease of the *Q*-factor with the resonant modes can be due to energy dissipation from dominant support loss, which can be defined [18]:

$$Q_{\mathrm{sup}} = \beta\left(\frac{L}{t}\right)^3 \tag{3}$$

where Q_{sup} is the support loss of *Q*-factor. The coefficient β decreases with the increase of vibration mode due to the larger vibration amplitude near to substrate.

The mass-sensitivity of the cantilever (γ_n) can be evaluated by the ratio of the resonant frequency shift, $\Delta f_{n,shift}$ to the added mass (Δm), which increases with an increasing eigenvalue (λ_n^2), and decreasing in both cantilever length (L) and cantilever width (w), which can be expressed as [19]:

$$\gamma_n = -\frac{\Delta f_{n,shift}}{\Delta m} = \frac{\lambda_n^2}{4\pi}\frac{1}{L^3 w}\frac{1}{0.236\sqrt{12}\tilde{\rho}}\sqrt{\frac{\tilde{E}}{\tilde{\rho}}} \tag{4}$$

where $\Delta f_{n,shift} = f_n - f_{n,\Delta m}$; f_n *and* $f_{n,\Delta m}$ are the resonant frequency of the piezoelectric cantilevers without and with added mass, respectively; $\tilde{E}$ and $\tilde{\rho}$ are the effective Young's modulus and effective density of the unimorph cantilever, respectively, that depends on the thickness fraction, Young's modulus and density of each of the individual layers. In this study, the added mass is a gold layer with dimension of 60×60 µm^2 and a mass of 4.878 ng, which was deposited on the tip of the piezoelectric cantilever by sputtering method.

Figure 7 shows the mass-sensitivity of the cantilevers as a function of the resonant mode for varying lengths (100–800 µm). It is shown that the mass-sensitivity of the piezoelectric cantilevers examined at the fundamental resonant modes increases from 0.1 Hz/pg to 37.6 Hz/pg when the length decreases from 800 to 100-µm. The measured results indicate that the mass-sensitivity of the piezoelectric cantilever exhibits the larger value for higher-order resonant mode. As an example, the mass-sensitivity of 800-

µm-long cantilever has value of 0.1 Hz/pg and 7.6 Hz/pg for the first resonant mode and sixth resonant mode, respectively. Thus, the piezoelectric cantilevers should be operated at the high resonant mode to gain the large value of mas-sensitivity, which is very important for mass-sensing applications.

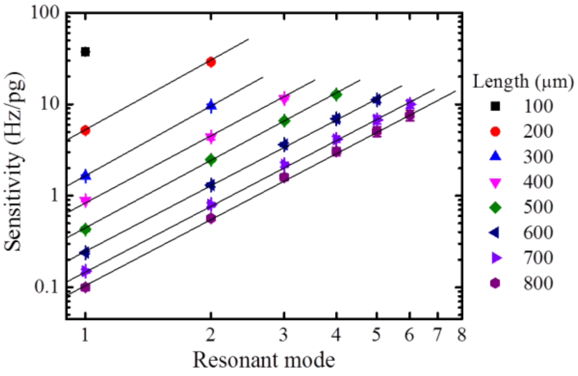

Figure 7. Relationships between resonant mode and cantilever length with mass-sensitivity.

4. Conclusion

In summary, we have shown that the piezoelectric microcantilevers can provide a very promising and powerful tool for accurate detection of small masses of materials. As mass sensors, the piezoelectric cantilevers in this study exhibit the mass-sensitivity on the order of pg/Hz (e.g. 0.027 pg/Hz or 37.6 Hz/pg for 100-µm-long cantilever). The experimental results indicate that the mass-sensitivity increases as the length of cantilever decreases, which can be predicted by the simple model. Moreover, the mass-sensitivity increases when the piezoelectric cantilevers operate at higher vibration resonant modes. However, the reduction of cantilever size is limited by microfabrication capability, and therefore, by working with higher vibration resonant modes, the longer piezoelectric cantilevers are still essential in the case of the larger surface area is required for mass sensing.

Acknowledgments

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