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Determining the Poisson's ratio of thin film materials using resonant method

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Abstract

In this study, a resonant technique to characterize the Poisson's ratio of thin film materials is proposed. The diagnostic micromachined cantilevers are fabricated using the thin film to be determined. The Poisson's ratio of the thin film can be determined after the bending and torsional vibration modes are measured. To obtain cantilevers with prismatic cross section, the micromachined beams were fabricated on the (1 1 1) silicon substrate. The primary advantage of this technique is that the error of Poisson's ratio due to the deviation of film thickness can be significantly reduced. In application of this technique, the Poisson's ratio of thermally grown silicon dioxide is measured to be 0.202 ± 0.021.

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Keywords: Poisson's ratio; Thin film materials; Bending and torsional vibration modes; Cantilever beams; (1 1 1) substrate

1. Introduction

In general, the material properties of thin film may not be the same as those of the bulk one. Moreover, the process by which the thin film is deposited affects the material properties. Thus, it is important to determine the mechanical properties of thin film materials in order to predict the performances of MEMS devices. Because thin films have thickness of the order of microns, the measurement methods used for bulk materials, such as the tensile test, are no longer appropriate. Presently, various testing techniques to determine the Young's modulus [1–3], residual stress [4,5] and coefficient of thermal expansion [6] of thin films using micromachined structures have been investigated.

The Poisson's ratio is used in almost all areas of stress analysis and structural dynamics. However, there are very few reports regarding the characterization of Poisson's ratio of thin film materials. In 1992, Vlassak and Nix employed the bulge test to measure the deflection of rectangular membrane under uniform pressure [7]. Thus, the Poisson's ratio can be determined indirectly after curve fitting of the measured results to a FEM model. Tabata used the same approach to determine the Poisson's ratio of thin film by modifying the FEM model [8]. Sharpe exploited the concept of the conventional tension test to determine the Young's modulus as well as Poisson's ratio of thin film materials directly [9]. In his experiment, the interferometric strain/displacement gage (ISDG) was used to measure the strain of specimen under tension stress. After measuring the lateral and axial strain of micro specimens, the Poisson's ratio is determined. Experimentally, the measurements in [7,8] are less complicated as compared to that in [9], so as to reduce the error caused by experiment. On the other hand, the test structures in [7,8] are more complicated to be well modeled, in which case errors arise from the simplifications or assumptions of the model. Moreover, all of these approaches are very sensitive to the film thickness. However, accurate measurement of the thickness of a micromachined structure is difficult, once the substrate or sacrificial layer has been removed. The M-test technique is a relatively simple technique, which determines the mechanical property of thin films by measuring the pull-in voltage of diagnostic structures [10]. The accuracy of M-test technique is influenced by the boundary conditions, residual gradient stress, and the film thickness. Moreover, the M-test technique is only applied for conductive materials.

In [11], the Poisson's ratio of materials was determined indirectly after the bending and torsional modes of a free-free beam were measured. This approach was modified, and then exploited to characterize the Poisson's ratio of thin films in the present study. To meet the requirements of

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design and fabrication processes, the diagnostic micromachined cantilevers were devised. Since the diagnostic micromachined cantilevers were fabricated using the thin film to be determined, this technique has the potential to be exploited for on-line characterization.

2. Analysis

The analysis intends to investigate the relation between the Poisson's ratio υ and the natural frequencies of a diagnostic micromachined cantilever. The Poisson's ratio υ of a homogenous and isotropic material is a function of the Young's modulus E and the shear modulus G [12]:

$$\upsilon = \frac{E}{2G} - 1 \tag{1}$$

According to the vibration analysis of a Euler–Bernoulli beam model [13], the Young's modulus E and the nth natural frequency of the bending vibration $(f_\mathrm{B})_n$ of a cantilever has the following relationship:

$$E = \frac{48\pi^2 \rho L^4 (f_\mathrm{B})_n^2}{(\beta_n L)^4 h^2} \tag{2}$$

where ρ is the density, L and h the length and thickness of cantilever, respectively. The parameter $(\beta_n L)$ is the nth eigenvalue of the governing equation for a clamp-free beam. In addition, the shear modulus G and the natural frequency of the first torsional vibration mode f_T of the cantilever has the following relationship [13,14]:

$$G = \frac{4\rho L^2 (b^2 + h^2) f_\mathrm{T}^2}{3ch^2} \tag{3}$$

where b is the width of cantilever and the constant c a coefficient that depends on the ratio b/h. From Eqs. (1)–(3), the relation between Poisson's ratio υ and natural frequencies $(f_\mathrm{B})_n$ and f_T of cantilever can be expressed as

$$\upsilon = \frac{18\pi^2 c L^2}{(\beta_n L)^4 b^2 (1 + (h/b)^2)} \left(\frac{(f_\mathrm{B})_n}{f_\mathrm{T}} \right)^2 - 1 \tag{4}$$

In short, the Poisson's ratio can be extracted from Eq. (4) after the dimensions L, b, h, and natural frequencies of bending and torsional mode, $(f_\mathrm{B})_n$, f_T, of cantilever were measured.

3. Experiment and results

In application of this technique, a wet thermal silicon dioxide film was measured as a case study. First, the diagnostic micromachined cantilevers were fabricated using the silicon dioxide film. After the natural frequencies of the diagnostic silicon dioxide cantilevers were measured, the Poisson's ratio of the film can be extracted from Eq. (4). In the experiment, the test micromachined cantilevers with length ranging from 100 to 200 µm were fabricated. The number of the measured specimens were more than 15 for each length. Since the proposed technique is very sensitive to the measured natural frequencies of the cantilever, two vibration test approaches were employed in this study.

3.1. *Specimen preparation on (1 0 0) and (1 1 1) substrate*

Firstly, the silicon dioxide with thickness about 1010 nm was thermally grown on a single crystal silicon wafer with (1 0 0) orientation at 1050 °C using the wet thermal oxidation process. Then, the silicon dioxide film was patterned as cantilevers using the photolithography technique and buffered HF. Finally, the silicon substrate that was below those cantilevers was removed anisotropically by 15% KOH etchant solution at 75 °C. The SEM photo of diagnostic micromachined cantilevers on (1 0 0) substrate after anisotropic etching is shown in Fig. 1a. In the experiment, the selectivity of the thermally grown silicon dioxide film and single crystal silicon substrate under the KOH solution is not infinite. Since the undercut is along the beam length, it took 50 min to fully remove the substrate under a 200 µm long cantilever. Thus, the change of the cross section as well as the thickness of the silicon dioxide beam by anisotropic bulk etching should not be ignored. As indicated by the dashed lines in Fig. 1b, the ideal cross section of the cantilever has a width b_0 and a thickness h_0 by assuming that the KOH etchant has infinite selectivity for the silicon substrate over the silicon dioxide film. In the real case, the cross section of the cantilever on an (1 0 0) oriented silicon wafer remains a rectangle, whereas its thickness varies linearly with the longitudinal location of the beam, as illustrated in Fig. 1b. Apparently, the natural frequencies of the cantilever for both bending and torsional modes will be influenced.

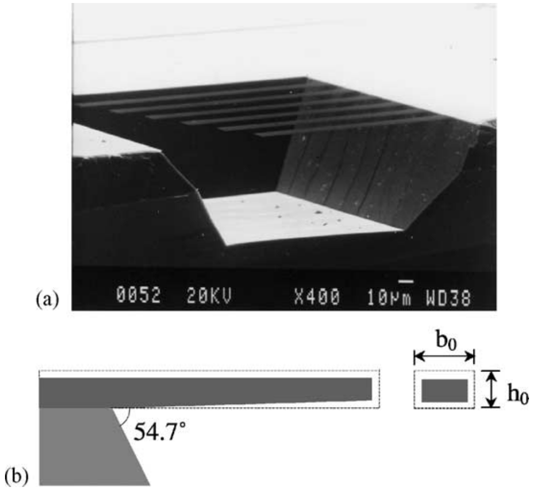

Fig. 1. Silicon dioxide micromachined cantilevers fabricated on (1 0 0) oriented silicon substrate: (a) SEM photograph, and (b) variation of the film thickness after anisotropic etch of KOH.

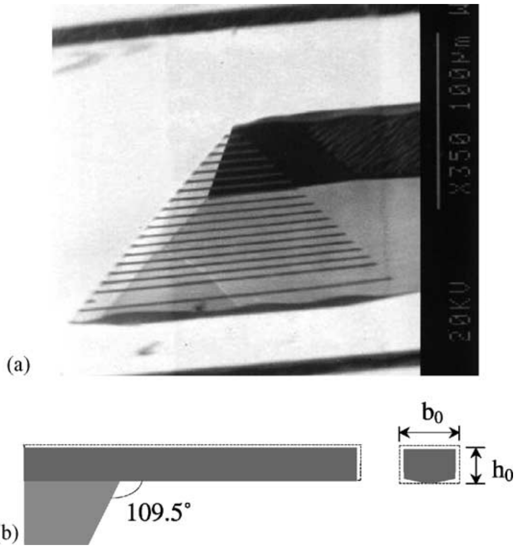

Fig. 2. Silicon dioxide micromachined cantilevers fabricated on (1 1 1) oriented silicon substrate for: (a) SEM photograph, and (b) variation of the film thickness after anisotropic etch of KOH.

The diagnostic micromachined cantilevers were also prepared on the (1 1 1) substrate to prevent the undercut effect [15]. The silicon dioxide with thickness about 1090 nm was thermally grown on (1 1 1) substrate. Unlike the (1 0 0) substrate process, the photoresist AZP 4620 was used as the mask to pattern the silicon dioxide film, and then the depth of released cavity of silicon substrate was defined by ICP dry etching. Finally, remove the silicon substrate anisotropically using the same etching solution. Since the undercut is along the beam width, the bulk etching time is determined by the width instead of the length of the cantilever. It took less than 15 min to fully remove the substrate under a 16 μm wide cantilever. The SEM photo in Fig. 2a shows the micromachined cantilevers on (1 1 1) substrate after anisotropic bulk etching. Since the undercut of a (1 1 1) wafer is along the width of the beam, the cantilever remains a prismatic beam. Moreover, the cross section of the beam on (1 1 1) wafer becomes a pentagon shown in Fig. 2b instead of a rectangle [15].

3.2. Experimental setup

Fig. 3 shows the schematic of experimental apparatus to characterize the dynamic response of micromachined cantilevers. The specimens were excited by an ultrasonic transducer underneath [16]. The dynamic response of the cantilevers was measured by the laser Doppler vibrometer (LDV) and recorded by the oscilloscope and the dynamic signal analyzer. In order to eliminate the air damping effect, the test sample was excited inside of a vacuum chamber. The air pressure inside this chamber can be reduced to 1 mTorr with a resolution of 0.1 mTorr.

The natural frequencies of the cantilevers can be excited through two different approaches. Firstly, the transducer is exploited to generate a pulsed bulk acoustic wave (BAW). The pulsed BAW performs as an impact hammer to excite the microstructures [17]. The BAW with a specific amplitude and time duration can excite all the vibration modes of a structure within a certain frequency range. A typical measured frequency spectrum on a 180 μm long and 16 μm wide cantilever on a (1 1 1) wafer by the impulse BAW test is shown in Fig. 4a. The peaks represent three bending modes $(f_B)_n$ and one torsional mode f_T, as indicated in Fig. 4a. The first bending mode of the cantilever was not excited since its frequency is beyond the energy bandwidth of the transducer. As a second approach, the ultrasonic transducer is exploited to generate a base harmonic excitation (BHE) on the micromachined cantilever. In other words, the BHE performs as a high frequency shaker to drive the microstructures. The microstructures will experience a large response when its natural frequencies coincident with the driving frequency of the ultrasonic shaker. Thus, the natural frequencies of the micromachined beam within a certain frequency range can be determined through the swept sine test. A typical measured frequency spectrum on a 180 μm long and 16 μm wide cantilever on a (1 1 1) wafer by the BHE test is shown in Fig. 4b. The peaks in Fig. 4b represent the first four bending modes $(f_B)_n$ and the first torsional mode f_T. For the BHE approach, the coupling of the dynamics between the micromachined beams and the ultrasonic shaker need to be

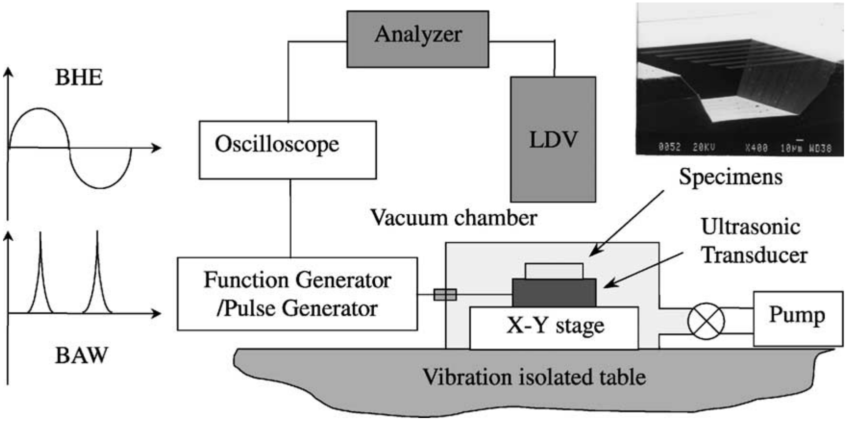

Fig. 3. The schematic of experimental apparatus.

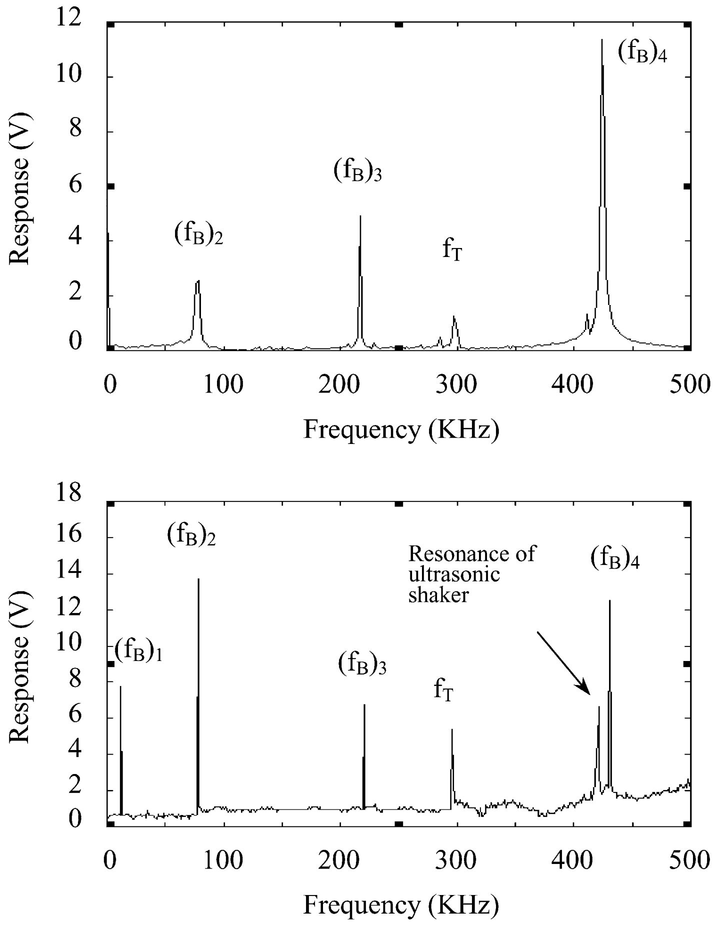

Fig. 4. Typical measured frequency response of test micromachined cantilever using: (a) BAW, and (b) BHE techniques.

considered. For instance, the fifth peak in Fig. 4b was caused by the resonance of the ultrasonic shaker.

3.3. Results

Fig. 5 shows the measured natural frequencies of bending vibration of a 180 μm long and 16 μm wide cantilever obtained from Fig. 4. The first four bending modes of this cantilever were ranging from 10.9 to 380.6 KHz. In addition,

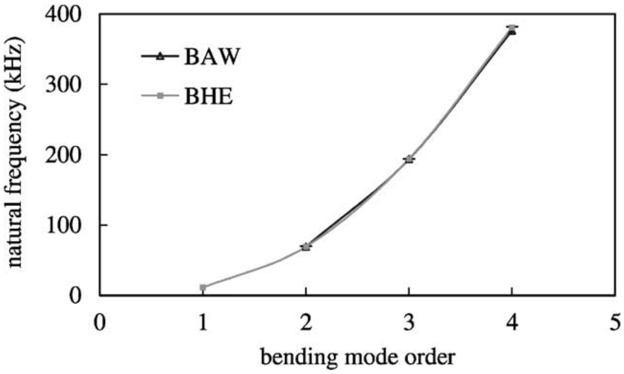

Fig. 5. The measured bending modes of micromachined cantilever on (1 1 1) Si substrate, which excited by BAW and BHE exciting techniques.

the measured torsional mode f_T was 261.8 KHz. Comparing with the results measured from the BAW and BHE approaches, their deviations at three different modes are all less than 1%.

After the natural frequencies of 200 μm long cantilevers were determined by the BHE approach, the Young's modulus, shear modulus, and Poisson's ratio can be extracted from Eqs. (2)–(4), respectively. The data points in Fig. 6a represent the Young's modulus determined by the first three bending modes of the 200 μm long cantilevers. Since the shear modulus was 23.70 ± 1.34 GPa, the Poisson's ratio determined by different bending modes is shown in Fig. 6b. As a result, the average Poisson's ratio of silicon dioxide film is 0.207 ± 0.001. The variation of the measured Poisson's ratio with the length of test beams was also investigated in this study. Fig. 7 indicates that the Young's modulus, shear modulus, and Poisson's ratio of the silicon dioxide film determined by the cantilevers with length ranging from 170 to 200 μm. As shown in Fig. 7a and b, the Young's modulus and shear modulus of silicon dioxide film are 56.69 ± 0.24 and 23.66 ± 0.38 GPa on average, respectively. Consequently, the Poisson's ratio of silicon dioxide film in Fig. 7c is 0.204 ± 0.008 on average. In summary, the average Poisson's ratio of the silicon dioxide film determined from

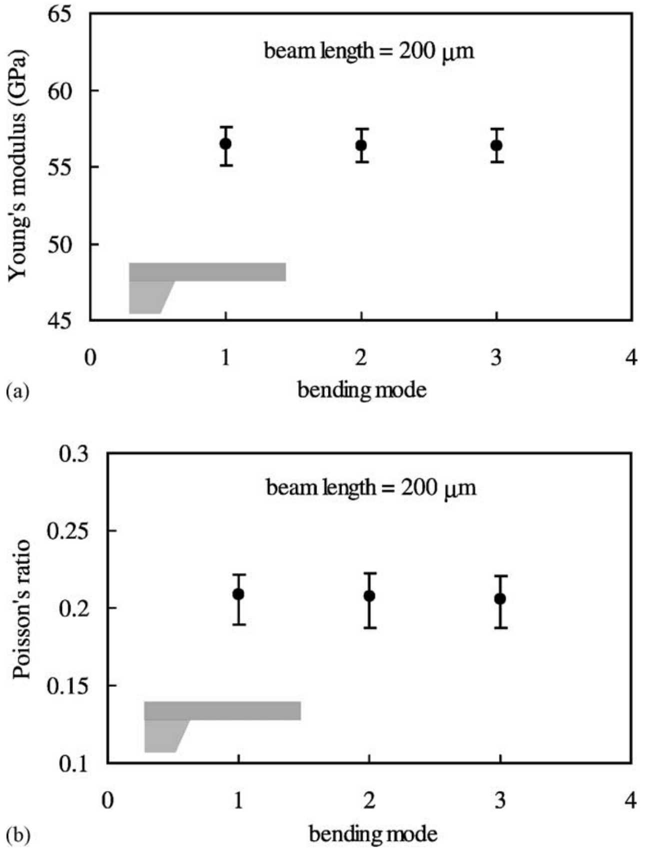

Fig. 6. (a) Young's modulus, and (b) Poisson's ratio of SiO_2 film that determined from the diagnostic beams on (1 1 1) Si substrate at the first three bending modes.

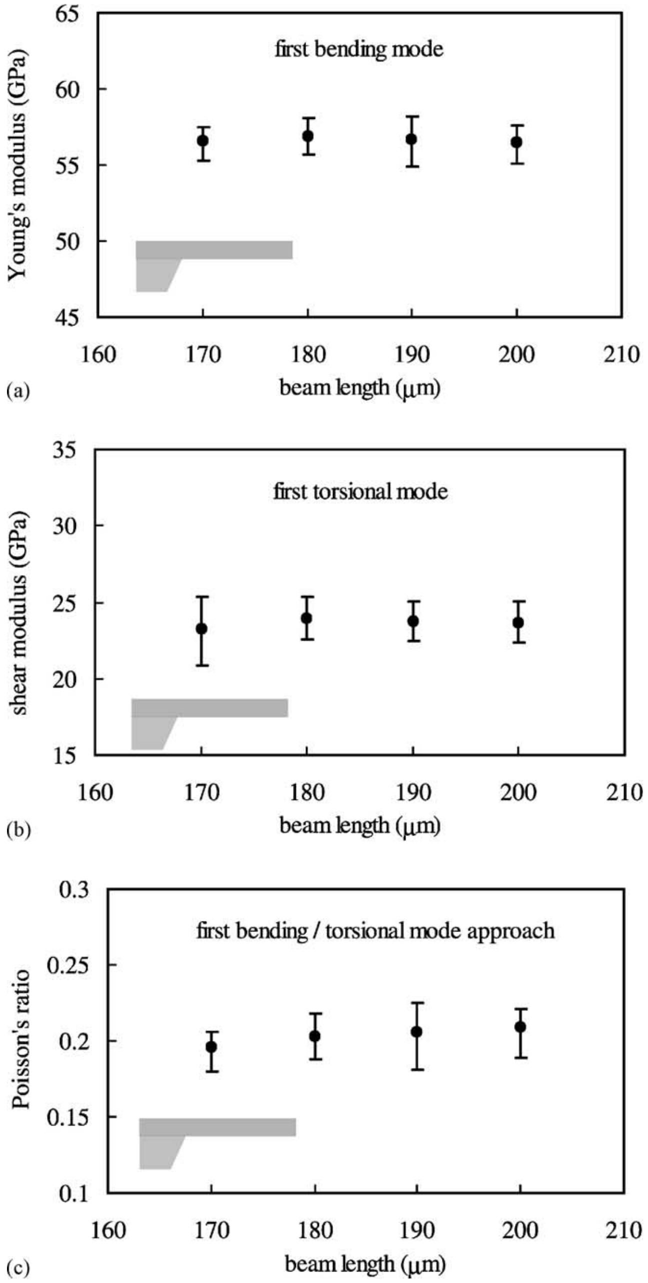

Fig. 7. (a) Young's modulus, (b) shear modulus, and (c) Poisson's ratio of SiO_2 film that determined from the diagnostic beams on (1 1 1) Si substrate for different beam lengths.

both different bending modes and different beam lengths of diagnostic cantilevers is 0.202 ± 0.021. Comparing with the existing report, the Poisson's ratio of silicon dioxide film, which extracted from microindentation test, is 0.17 [18].

4. Discussion

The proposed technique is sensitive to the measured natural frequencies of the cantilever. The air damping effect will cause a frequency shift about more than 1% when the ambient pressure increases from 5 mTorr to 1 atm. According to Eq. (4), a more than 4% error in Poisson's ratio will occur. Consequently, the measurement has to be conducted inside a vacuum chamber to avoid the air effect.

It is critical for this technique to satisfy the assumption of having a uniform cross section along the beam length. Due to the undercut mechanism, the cross section of the cantilever on (1 0 0) wafer will vary with the beam length, as indicated in Fig. 1b. The frequencies of the bending and torsional modes will be shifted. Moreover, the higher bending modes will experience a larger deviation. Accordingly, the Poisson's ratio extracted from the natural frequencies of the cantilever will vary with the vibration modes. The solid dots in Fig. 8 show the Poisson's ratio extracted from the first four bending modes of a 200 μm long and 16 μm wide cantilever on a (1 0 0) wafer. It is obtained that the Poisson's ratio associated with different vibration modes varies from 0.22 to 0.33. A finite element model was also established in this study to analyze the variation of the Poisson's ratio with the vibration modes. As indicated by the hollow dots in Fig. 8, the results predicted from the finite element analysis of a trapezoid beam model shows good agreement with the measurement. The dimensions of FEM model were based on the actual dimensions of 200 μm long and 16 μm wide beam. Since

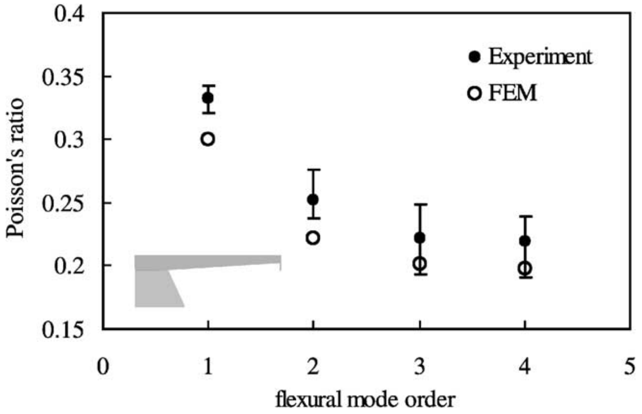

Fig. 8. Poisson’s ratio of SiO_2 film that determined from the diagnostic beams on (1 0 0) Si substrate at the first four bending modes.

the etching rate of wet thermal oxide under KOH solution was 3.2 nm/min in this study, the thickness of the beam at the free end and fixed end are 0.69 and 0.85 µm, respectively. In addition, the Young’s modulus and Poisson’s ratio employed in the FEM model were 70 GPa and 0.17, respectively. Therefore, the diagnostic cantilever on a (1 0 0) substrate is not appropriate for the proposed approach. On the contrary, the cantilever on a (1 1 1) wafer has uniform cross section after bulk etching, hence, the Poisson’s ratio measured from the diagnostic cantilever on (1 1 1) wafer will not vary with the vibration modes. This has been demonstrated by the measured results in Fig. 6b.

The dimensions of diagnostic cantilevers are critical parameters to influence the characterization of Poisson’s ratio. In general, it is difficult to precisely measure the thickness of suspended micromachined structures after bulk silicon etching. Consequently, the uncertainty of the thin film thickness is the primary error source for the existing approaches when extracting the material properties from diagnostic micromachined structures. The width b and thickness h of the diagnostic cantilevers employed in this study is approximately one order of magnitude difference. According to Eq. (4), the error in υ due to the deviation of h can be significantly reduced. For instance, the error in υ for a 1 µm thick and 20 µm wide beam is only 0.27% when the deviation of the film thickness, h is 10%.

5. Conclusion

The Poisson’s ratio of thin film materials is a very important mechanical property for stress analysis and structural dynamics. However, it is difficult to be determined accurately and conveniently so far. In this study, the Poisson’s ratio of thin films can be extracted indirectly from the measured natural frequencies of the bending and torsional vibration of micromachined cantilevers. The advantage of this technique is to determine the Poisson’s ratio in a single measurement through a ready fabricated and modeled micromachined cantilever. Because the approach is very simple, it can be applied as a supplement to the other Poisson’s ratio measurement techniques. This technique is very sensitive to the measured vibration frequencies of the diagnostic cantilever. Consequently, the test has to be conducted inside a vacuum chamber. Moreover, the diagnostic cantilever has to be fabricated on the (1 1 1) silicon substrate.

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Biographies

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