

## Lead zirconate titanate films for $d_{33}$ mode cantilever actuators

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### Abstract

Piezoelectric  $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$  (PZT 52/48) cantilever actuators that operate in the  $d_{33}$  mode were fabricated using surface micromachining techniques. The cantilevers are composed of a PZT thin film on a low stress silicon nitride ( $\text{Si}_x\text{N}_y$ ) support buffered by a layer of  $\text{ZrO}_2$ . Au/Cr interdigitated (IDT) electrodes were deposited on the PZT film surface, so that the cantilever could be poled and actuated in the  $d_{33}$  mode. The PZT deposited on the  $\text{ZrO}_2/\text{Si}_x\text{N}_y/\text{Si}$  stack is well-crystallized into the perovskite phase and is randomly oriented. It exhibits good ferroelectric behavior with a remanent polarization of  $26 \mu\text{C}/\text{cm}^2$ . The cantilevers have a width of  $100 \mu\text{m}$  and lengths between  $130$  and  $280 \mu\text{m}$ . The fundamental resonance frequencies are in the range of  $6.1$ – $29.6$  kHz. When actuated with an applied electric voltage of  $100$  V, the  $280 \mu\text{m}$  long cantilever exhibits a large downward tip displacement of  $30 \mu\text{m}$ .

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### 1. Introduction

Recently, ferroelectric lead zirconate titanate (PZT) thin films have attracted attention for microelectromechanical system (MEMS) devices, such as micro-actuators and micro-sensors because they have desirable properties, such as high piezoelectric coefficients [1]. A number of surface micromachined actuators utilizing PZT films have been reported [2]. Typically, the actuators entail a cantilever beam consisting of a thin structural material supporting a PZT film sandwiched between top and bottom electrodes as shown in Fig. 1a. The in-plane strain  $x_1$  in the PZT, induced by an external electric field  $E_3$  normal to the plane, is expressed by the converse piezoelectric effect:

$$x_1 = d_{31}E_3 \quad (1)$$

where  $d_{31}$  is the transverse piezoelectric coefficient, typically a negative number. When a voltage is applied to the top and bottom electrodes, the PZT film contracts laterally for  $E_3$  parallel to the remanent polarization ( $P_r$ ) of PZT,

which makes the beam bend up. PZT expands laterally with  $E_3$  antiparallel with  $P_r$ , bending the beam down. However, the polarization state changes when  $E_3$  is large compared to the coercive field of PZT. The large  $E_3$  repoles the PZT, again making  $E_3$  parallel with  $P_r$ , which makes the beam bend up again. Thus, this kind of cantilever can produce large upward bending displacements, actuated by the piezoelectric  $d_{31}$  effect of the PZT.

When an interdigitated (IDT) electrode is deposited on the top of PZT as shown in Fig. 1b, both the applied electric field and the polarization are largely in the plane of the PZT film. The induced in-plane strain  $x_3$  resulting from an in-plane electrical field  $E_3$  is also expressed by the converse piezoelectric effect:

$$x_3 = d_{33}E_3 \quad (2)$$

where  $d_{33}$  is the longitudinal piezoelectric coefficient of PZT, typically a positive number. The transverse piezoelectric strain is converted into cantilever bending, so the beam is actuated by the piezoelectric  $d_{33}$  effect of the PZT. It is well known that the magnitude of the  $d_{33}$  coefficient of PZT is about twice the  $d_{31}$  coefficient, thus cantilever beams actuated by the  $d_{33}$  effect are expected to produce substantial deflections [3]. Fig. 1b also shows that  $d_{33}$  mode cantilevers are primarily downward-moving cantilevers since the PZT

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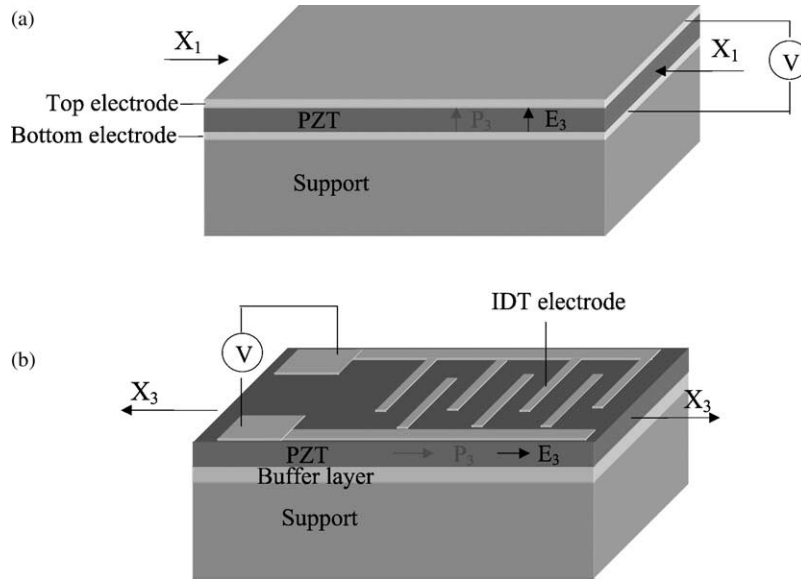


Fig. 1. Schematic diagram of cantilever actuated by (a)  $d_{31}$  mode and (b)  $d_{33}$  mode (they are not drawn to scale).

film expands in-plane when  $E_3$  is applied parallel to  $P_r$ ; again note that the magnitude of  $E_3$  is limited by the coercive field value when applied antiparallel to  $P_r$ . Downward-moving cantilevers have an advantage in applications, such as some local probe measurements or in an integrated microswitch where the actuator (cantilever beam) and signal lines are located on the same wafer [4]. The spacing of the IDT electrodes can be varied lithographically, depending on the desired actuation voltage. This is another major advantage of  $d_{33}$  mode actuation since the thickness of the PZT film does not have to be changed to achieve a different capacitance (impedance matching) or actuation voltage.

However, few studies have been devoted to the fabrication and characterization of  $d_{33}$  mode cantilevers [3,5]. We have been investigating the  $d_{33}$  mode cantilevers for

microswitches [6]. In this work, PZT  $d_{33}$  mode cantilevers were fabricated using surface micromaching techniques. Low stress silicon nitride ( $Si_xN_y$ ) was used as the support layer. High quality PZT films cannot be deposited directly on  $Si_xN_y$ , so a buffer layer is needed to prevent reaction and interdiffusion.  $ZrO_2$  has previously been shown to be a good buffer layer between PZT and a thermally oxidized silicon substrate [7]. Thus,  $ZrO_2$  thin films were also explored as a buffer layer between the PZT and the low stress  $Si_xN_y$  in this work. Because the properties of PZT depend on the substrate, the structure and ferroelectric behavior of the PZT deposited on the multilayer  $ZrO_2/Si_xN_y/Si$  substrate were characterized. This paper also presents the fabrication process for the  $d_{33}$  mode cantilevers and results on the resonance frequency and deflection. CoventorWare (Coventor,

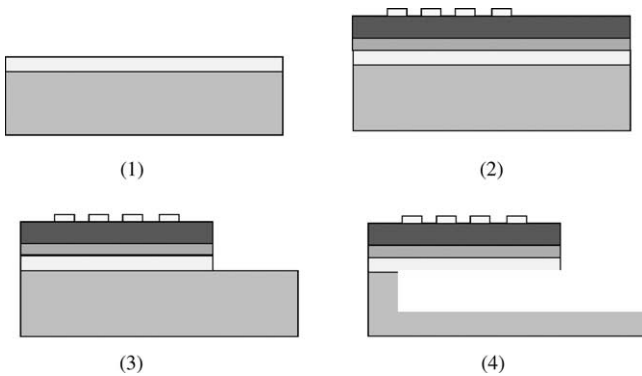


Fig. 2. Fabrication flow for  $d_{33}$  mode cantilevers. (1) Silicon wafer with  $0.5\ \mu\text{m}$  low stress  $Si_xN_y$ . (2) A  $ZrO_2$  buffer layer was deposited on  $Si_xN_y$  and then PZT was deposited by a sol-gel technique. The top interdigitated Au/Cr electrode was evaporated on the PZT and patterned by lift-off. (3) PZT and  $ZrO_2$  were patterned into a cantilever-shaped structure using ion-milling, and  $Si_xN_y$  was patterned using RIE. (4) Cantilever was released by etching the underlying silicon using vapor-phase  $XeF_2$ .

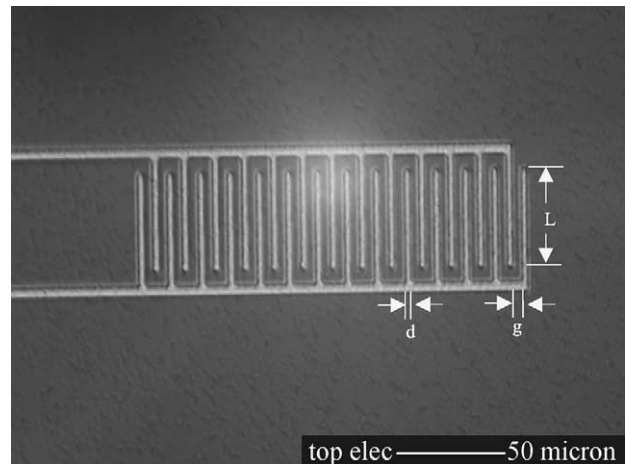


Fig. 3. Lift off Au/Cr IDT electrode on the  $280\ \mu\text{m}$  long cantilever with finger width,  $d$ , of  $3\ \mu\text{m}$ , electrode gap width,  $g$ , of  $6\ \mu\text{m}$ , electrode length,  $L$ , of  $60\ \mu\text{m}$  and finger number,  $N$ , of 27.

Cary, NC), which is a MEMS design and analysis software based on finite element analysis, was used to simulate the resonance frequency of the cantilevers and the results are compared with the experimental ones.

## 2. Experimental procedure

### 2.1. $ZrO_2$ buffer layer and PZT piezoelectric film

$ZrO_2$  films were deposited by a sol-gel method onto low stress silicon nitride  $Si_xN_y$ -coated silicon substrates. Zirconium *n*-propoxide was first added to 2-methoxyethanol (2-MOE), and the mixture was refluxed at 110 °C for 2 h under a dry Ar atmosphere. Following the reflux step, the solution was vacuum distilled at 120 °C. Then, 10% by volume of acetylacetone was added, and a 0.45 molar  $ZrO_2$  solution was obtained. The solution was injected onto the substrate through a 0.2  $\mu m$  filter and was then spin-coated

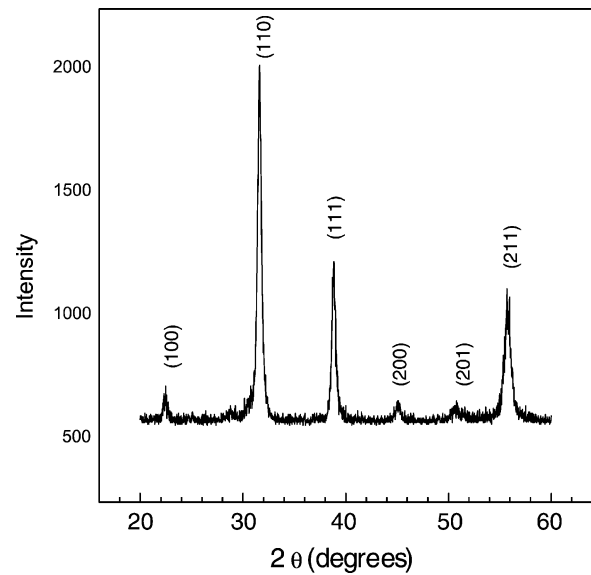


Fig. 4. XRD pattern of PZT deposited on  $ZrO_2/Si_xN_y/Si$  substrate.

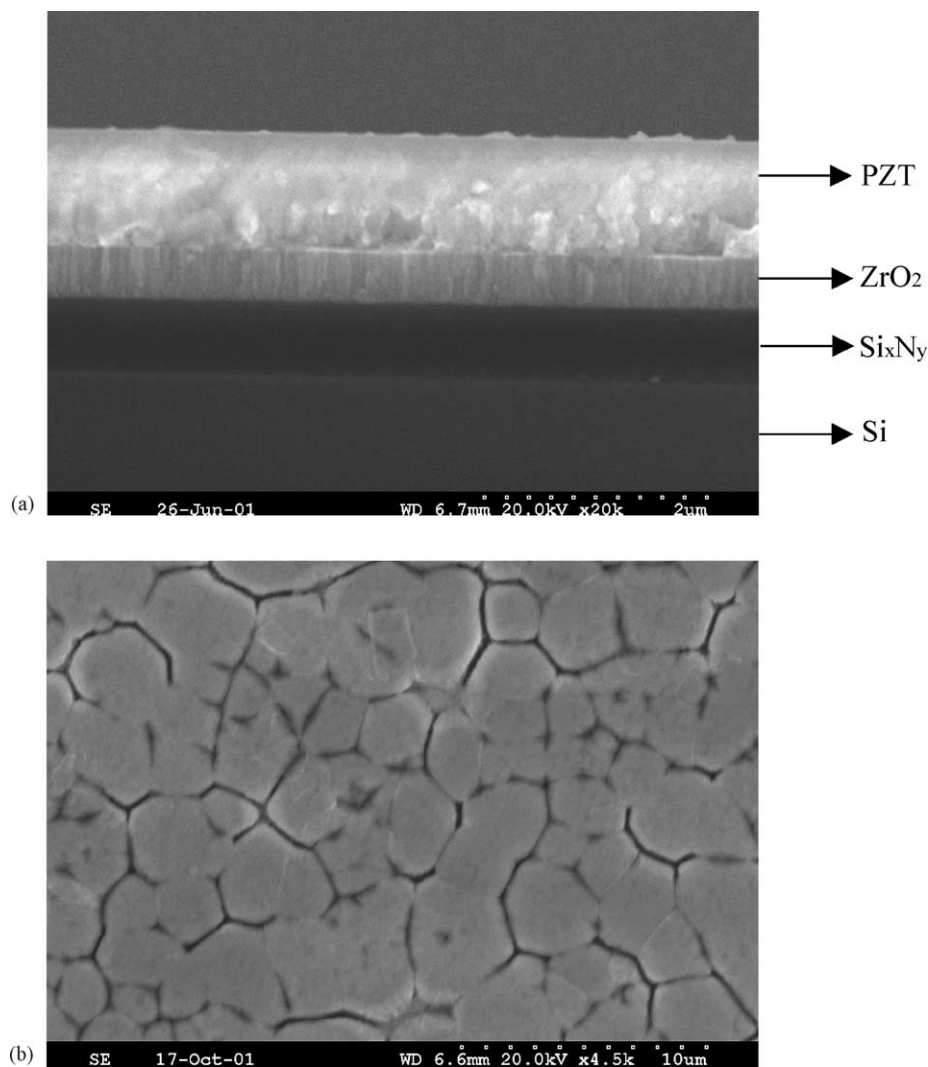


Fig. 5. SEM of PZT deposited on  $ZrO_2/Si_xN_y/Si$  substrate: (a) cross-section and (b) surface.

at 3000 rpm for 30 s. After deposition, each layer was subjected to pyrolysis at 300–450 °C to drive out the solvent and decompose organic compounds, and then annealed at 650 °C for 60 s by rapid thermal annealing (RTA, Heatpulse 610, A.G. Associates, San Jose, CA). The thickness of each layer was about 50 nm. A 300 nm thick  $\text{ZrO}_2$  was obtained by repeating the process six times. Finally, the film was fired in a furnace at 700 °C for 1 h.

PZT films were then deposited on the  $\text{ZrO}_2$  by a sol-gel process described previously [8]. Briefly, lead acetate trihydrate, titanium *iso*-propoxide and zirconium *n*-propoxide were used as starting materials, and 2-MOE was used as the solvent. Twenty percent excess lead was batched in

the  $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$  solution. Layers of approximately 0.2  $\mu\text{m}$  thickness were deposited from 0.7 M solution by spin coating at 1500 rpm for 30 s. Each layer was pyrolyzed at 300–450 °C on a hot plate and then crystallized at 700 °C by RTA. A total of four layers were deposited and crystallized to obtain a 0.8  $\mu\text{m}$  thick PZT film.

## 2.2. $d_{33}$ mode cantilever actuators

Fig. 2 shows the fabrication process flowchart for PZT cantilevers actuated in the  $d_{33}$  mode. A 0.5  $\mu\text{m}$  thick low stress  $\text{Si}_x\text{N}_y$  layer, deposited on the silicon substrate by low-pressure chemical vapor deposition (at the University

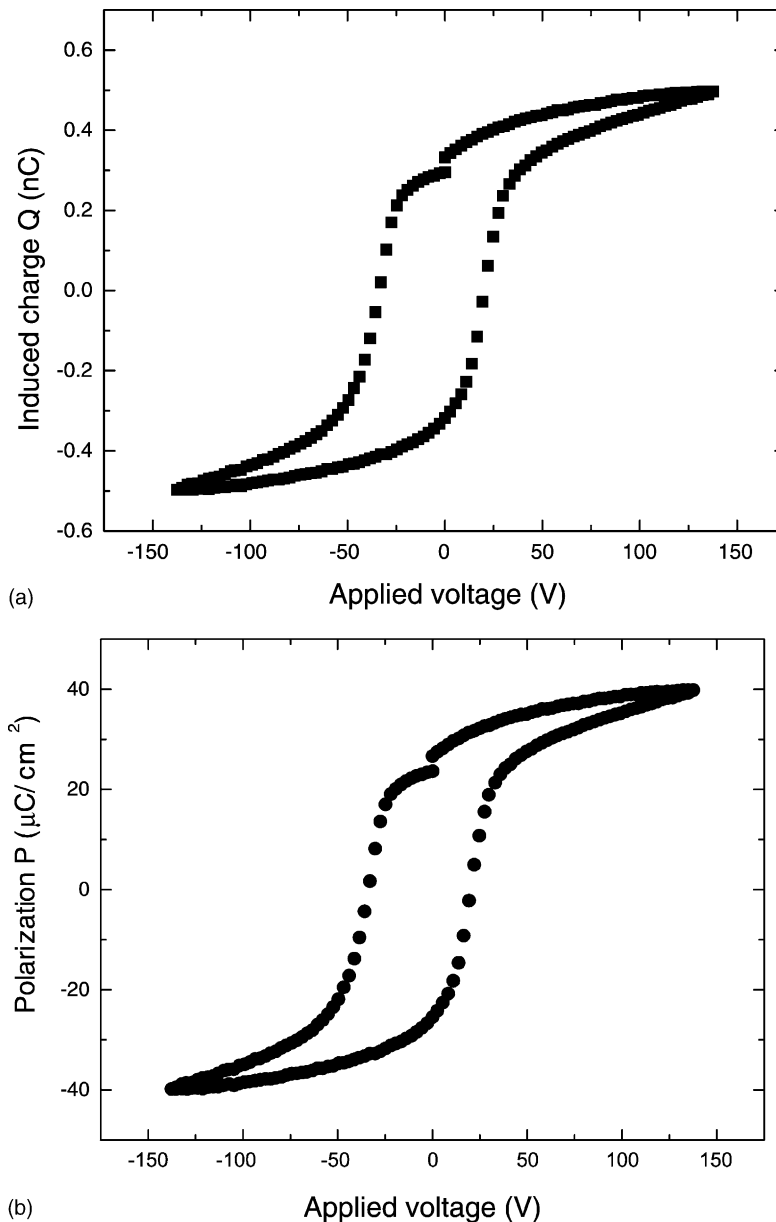


Fig. 6. (a)  $Q$ - $V$  loop of PZT with IDT electrodes, and (b)  $P$ - $V$  loop of PZT with IDT electrodes (the data in (b) were calculated from (a)/ $A$  where  $A$  is given in Eq. (3)).

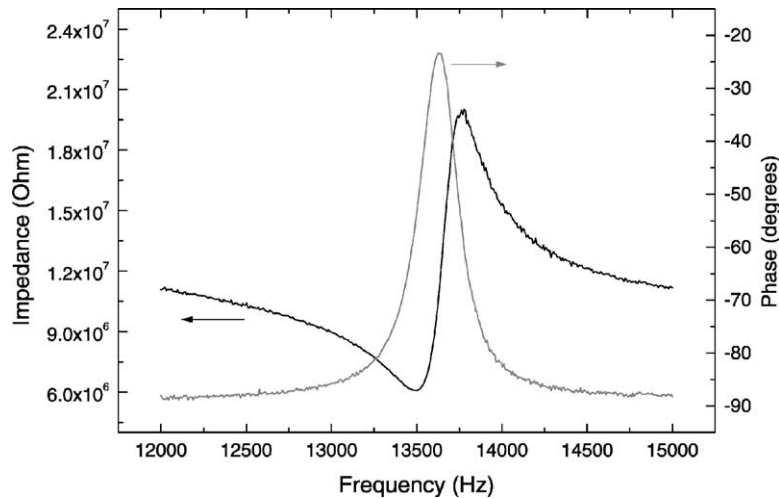


Fig. 7. Measured resonance curve for the  $d_{33}$  cantilever with length of 180  $\mu\text{m}$  and width of 100  $\mu\text{m}$ .

of Minnesota), was used as the support layer. A 0.3  $\mu\text{m}$   $\text{ZrO}_2$  film was used as the buffer layer between the PZT and the  $\text{Si}_x\text{N}_y$ . Following the PZT deposition, Au/Cr was evaporated on the PZT and patterned by a lift off process into IDT electrodes with finger widths of 3  $\mu\text{m}$  and a gap width of 6  $\mu\text{m}$ . Layer thicknesses of 100 and 10 nm were used for Au and Cr, respectively. Fig. 3 is an optical micrograph of the IDT electrode by lift off on the 280  $\mu\text{m}$  long cantilever.

PZT and  $\text{ZrO}_2$  were patterned into cantilever-shaped structures using ion-milling. Ion-milling was performed in an Oxford series 300 dual beam system with a 15 cm etch source.  $\text{Si}_x\text{N}_y$  was patterned by a plasma reactive ion etching (RIE) system with  $\text{CF}_4$  and  $\text{O}_2$ . Finally, the cantilevers were released by etching the underlying silicon using vapor-phase  $\text{XeF}_2$ . By using this process, cantilevers with a width of 100  $\mu\text{m}$  and various lengths from 80 to 280  $\mu\text{m}$  were fabricated.

### 2.3. Characterization

The structure of the as-deposited PZT film was determined using X-ray diffraction (XRD, XDS2000, Scintag) with Ni filtered  $\text{Cu K}\alpha$  radiation. X-ray diffraction patterns were recorded at a rate of  $2^\circ/\text{min}$  in the range of  $2\theta$  from 20 to  $60^\circ$ . The thicknesses of the films were measured using a surface profiler (Alpha-step 500, Tencor Instruments). A scanning electron microscope (SEM; Hitachi, S-3500N, Japan) was used to inspect the films and observe their morphology. The high field ferroelectric hysteresis properties were characterized using a RT66A (Radiant Technology, Albuquerque, NM) ferroelectric test system with an amplifier. An impedance analyzer (HP4194A, Hewlett-Packard) was used to measure the fundamental resonance frequency, and a Zygo (Zygo New View 100) interferometer was used to determine tip deflection.

### 3. Results and discussion

An XRD pattern of a PZT film deposited on a  $\text{ZrO}_2$  buffer layer is shown in Fig. 4. It can be seen that the PZT film is well-crystallized into the perovskite phase. The (1 1 0) peak exhibits the highest intensity, suggesting that the crystallites in the film are approximately randomly oriented.

Good adhesion is observed in the PZT/ $\text{ZrO}_2$ / $\text{Si}_x\text{N}_y$ / $\text{Si}$  multilayer from the cross section of the SEM micrograph as shown in Fig. 5a. The surface SEM micrograph (Fig 5b) shows the PZT has an average lateral grain size of about 3  $\mu\text{m}$ . This was confirmed using AFM measurements.

Fig. 6a is an induced charge–applied voltage ( $Q$ – $V$ ) hysteresis loop from the IDT electrodes of the 280  $\mu\text{m}$  long cantilever. Assuming the electric field is uniform across the electrode gap, the area ( $A$ ,  $\text{cm}^2$ ) of the PZT capacitance with

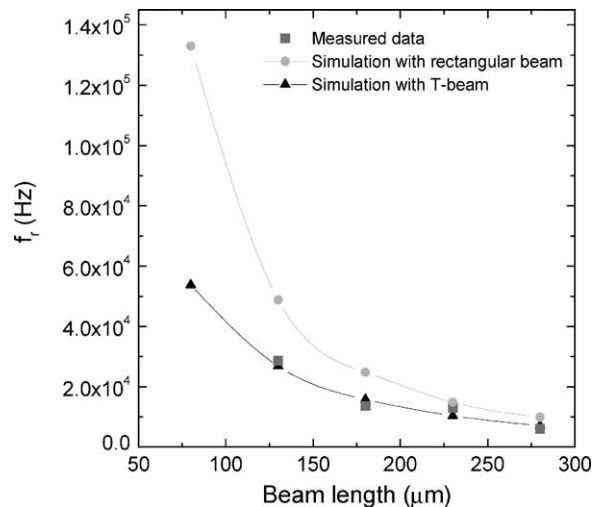


Fig. 8. Measured and simulated resonance frequencies as a function of cantilever length.

IDT electrodes can be calculated [3].

$$A = Lt(N - 1) = 12.5 \times 10^{-6} \quad (3)$$

where  $L$  ( $=60 \mu\text{m}$ ) is the length of the electrode as shown in Fig. 3,  $t$  ( $=0.8 \mu\text{m}$ ) is the PZT thickness, and  $N$  ( $=27$ ) is the number of IDT fingers. With this approach, the  $Q$ – $V$  loop can be converted to a polarization-field ( $P$ – $V$ ) loop ( $P = Q/A$ ) as shown in Fig. 6b. A remanent polarization of  $26 \mu\text{C}/\text{cm}^2$  is obtained from the loop, which is close to the value for PZT films deposited on platinum bottom electrodes for  $d_{31}$  actuation [8].

The PZT was poled at room temperature for 15 min by applying 100 V to the IDT electrodes. Subsequently, the electromechanical resonances of the released cantilevers were investigated by measuring the impedance of the PZT actuator as a function of frequency. As an example, Fig. 7 shows the measured result for the  $180 \mu\text{m}$  long cantilever, for which a fundamental resonance frequency,  $f_r$ , of 13.6 kHz is obtained.

In Fig. 8 the measured resonance frequencies for the various cantilevers are plotted as a function of the beam length.  $f_r$  decreases as beam length increases. CoventorWare was used to simulate the resonance frequency results. The materials parameters of each layer used in the simulation are listed in Table 1. As a first approximation, a rectangular beam

Table 1

Materials parameters used in the simulation of resonance frequency

Materials	Density (kg/m <sup>3</sup> )	Elastic constant (GPa)	Poisson's ratio
Gold [9]	19300	57	0.35
PZT [10]	7400	101	0.30
ZrO <sub>2</sub> [11]	6000	160	0.27

shape (Fig. 9a) was used. As seen in Fig. 8, the simulated resonances were higher than the measured ones. Realizing that the Si underneath the beam attachment was also etched out in the cantilever release process (Fig. 10) because XeF<sub>2</sub> is an isotropic silicon etch, a T-shaped beam (Fig. 9b) was also used in simulations. The width of the undercut for the T-shaped resonator was assumed to be  $50 \mu\text{m}$  (half of the beam width). The simulations for this geometry agree well with the measurements.

To observe the bending, the cantilevers were put in the SEM chamber and an external dc voltage  $V$  was applied. Downwards bending is observed as shown in Fig. 10. This result corresponds to the analysis in the introduction. The magnitudes of the bending displacement were measured using the Zygo interferometer. Fig. 11 shows the measured tip displacement as function of the cantilever length with an applied electric voltage of 100 V (the polarization of PZT

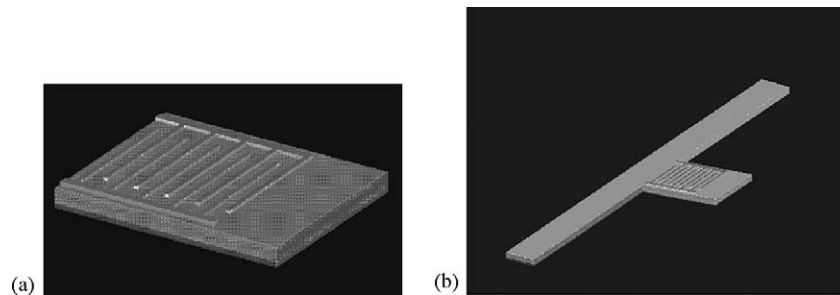


Fig. 9. (a) Rectangular beam shape and (b) T-beam shape used to simulate resonance frequency of the  $d_{33}$  cantilevers.

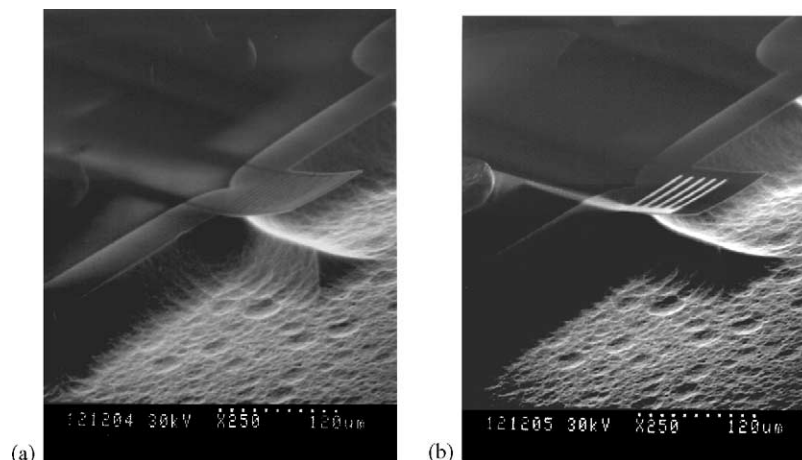


Fig. 10. SEM of the cantilever added external electric voltage: (a)  $V = 0$  and (b)  $V = 50$  V.

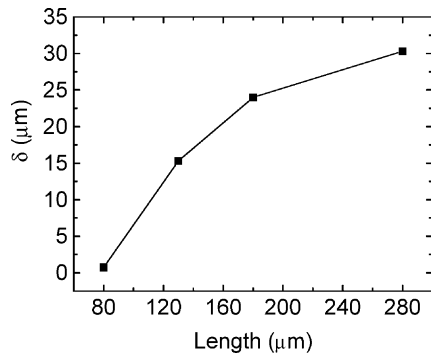


Fig. 11. Dependence of tip displacement on the cantilever length (applied voltage of 100 V).

is larger than its residual polarization in this condition because the applied field is larger than its coercive field). The displacement increases with beam length. The largest displacement of 30  $\mu\text{m}$  is obtained at 100 V for the 280  $\mu\text{m}$  long beam.

#### 4. Conclusions

A PZT/ZrO<sub>2</sub>/Si<sub>x</sub>N<sub>y</sub> stack has been used to fabricate  $d_{33}$  mode piezoelectric cantilevers. Sol-gel derived ZrO<sub>2</sub> is an acceptable buffer layer between PZT and Si<sub>x</sub>N<sub>y</sub>. The PZT has the perovskite structure, with approximately random orientation, and an average grain size of about 3  $\mu\text{m}$ . Good ferroelectric behavior, with a remanent polarization of 26  $\mu\text{C}/\text{cm}^2$ , is also observed in the PZT film.  $d_{33}$  mode cantilevers with a width of 100  $\mu\text{m}$  and lengths from 80 to 280  $\mu\text{m}$  have been successfully fabricated using surface micromachining. The resonance frequencies of the cantilevers with lengths from 130 to 280  $\mu\text{m}$  have been measured in the 6.1–28.6 kHz range, which agree with the simulations for T-shaped cantilevers. SEM observation confirms that the  $d_{33}$  cantilevers are downward bending ones. A tip displacement of 30  $\mu\text{m}$  has been measured in the cantilever with length of 280  $\mu\text{m}$ . The study of the displacement versus the magnitude and frequency of the applied field is in progress. The largest displacement is expected at an ac field with cantilever's resonance frequency.

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