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The wafer flexure technique for the determination of the transverse piezoelectric coefficient (d_{31}) of PZT thin films

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Abstract

This paper describes a simple and inexpensive method for evaluating the transverse piezoelectric coefficient (d_{31}) of piezoelectric thin films. The technique is based upon the flexure of a coated substrate which imparts an ac two-dimensional stress to the piezoelectric film. The surface charge generated via the mechanical loading is converted to a voltage by an active integrator. Plate theory and elastic stress analyses are used to calculate the principal stresses applied to the film. The d_{31} coefficient can then be determined from knowledge of the electric charge produced and the calculated mechanical stress. For 52/48 sol–gel lead zirconate titanate (PZT) thin films, the d_{31} coefficient was found to range from -5 to -59 pC/N and is dependent on poling field.

Keywords: Transverse piezoelectric coefficient; Thin films; PZT

1. Introduction

The design and development of novel microelectromechanical systems (MEMS) which utilize piezoelectric thin films [1,2] require explicit knowledge of the material's longitudinal (d_{33}) and transverse (d_{31} or d_{32}) piezoelectric coefficients. When prepared in bulk ceramic form, piezoelectric coefficients are characterized by numerous methods, the most common of which are resonance and dynamic load techniques (Belincourt meter). Those techniques, however, are inadequate for the piezoelectric characterization of thin film materials and for that reason a number of alternative techniques (e.g., laser beam interferometry and normal load methods) have been proposed [3,4].

Laser interferometers are the most well-established method for the characterization of both the longitudinal and transverse piezoelectric coefficients of piezoelectric thin films [3,5–7]. Interferometers can be configured as either single- or double beam setups and are based upon the interference of monochromatic laser light in response to a piezoelectrically-induced strain (which results in a change of the optical path length). The wavelength of the laser and the intensity of the interference signal produced are input to a simple mathematical model from which the electric-field induced strains are determined. The ultimate resolution of interferometric technique(s) is limited by extraneous displacements (e.g., sample flexure, thermal drift) and environmental noise (electrical and mechanical) to 10^{-4} Å for the best double-beam instruments [3] and 10^{-5} Å for certain single beam instruments [7]. This method requires careful optical alignment, meticulous operation, and in the case of a d_{31} measurement, appropriate sample preparation (cantilever beam construction).

There are alternatives to interferometric characterization and reports have been published on a number of designs which utilize the direct piezoelectric effect. The normal load method, as was described in the work of Lefki and Dormans [4], is analogous to the common Belincourt meter; however, the measured d_{33} values (400 pC/N for undoped sol–gel PZT with compositions near the morphotropic phase boundary) appear improbable in light of the limited twin wall motion in PZT thin films [8,9]. Transverse piezoelectric characterization (d_{31}) has also been attempted [10] and work was based upon the controlled bending of a small cantilever beam. Reasonable d_{31} values were obtained [11]; however, the necessary construction of cantilever test samples is a drawback to the technique.

The wafer flexure technique described in this paper is an alternative method for the characterization of the transverse piezoelectric coefficient (d_{31}) which eliminates a number of the complexities associated with other designs. Experiments conducted have produced d_{31} values comparable to those measured with more established methods (e.g., interferometric). However, the speed with which the measurement is

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made, the relative cost of the hardware, and the utilization of complete silicon substrates makes the wafer flexure technique an attractive alternative for the d_{31} characterization of thin films.

2. Principle of operation

The wafer flexure technique for measuring d_{31} is based upon the direct piezoelectric effect. The controlled bending of a PZT coated substrate imposes a planar two-dimensional mechanical stress upon the film. The strained film responds by developing a surface charge which is monitored electronically. The principle stresses applied to the film are then calculated using small deflection plate theory [12] and the piezoelectric coefficient is determined as

$$D_3 = d_{31}(\sigma_1 + \sigma_2) \qquad (1)$$

where d_{31} is the transverse piezoelectric coefficient (C/N), D_3 is the induced dielectric displacement (C/m^2), and σ_1 and σ_2 are the principal stresses applied to the film (N/m^2).

3. Experimental design

The wafer flexure apparatus consists of three discrete components (see Fig. 1): (1) the uniform pressure rig, (2) the charge integrator, and (3) the peripheral electronics. Each of these components will be described in the subsections which follow.

3.1. Uniform pressure rig

The uniform pressure rig consists of two aluminum parts, the housing and the retention ring, between which is placed a 3 in. (76.2 mm) test wafer. The inner radius of both portions measures 1.25 in. (31.75 mm) and at points of contact with the sample, the aluminum has been polished to 5 μm roughness (to improve the pressure seal and to reduce the risk of substrate fracture). The pneumatic pressure in the cavity behind the wafer is oscillated periodically with a 60 cm^3 plastic syringe. Pressure changes are monitored with a piezoresistive pressure transducer (Omega PX236, 30 psig full scale) excited and monitored with an EG&G 7260 lock-in amplifier. Fig. 2 shows a blown-up schematic of the design.

3.2. Charge integrator

The charge integrator used to monitor the change in the film's dielectric displacement as a function of mechanical stress is shown in Fig. 3. Charge from the piezoelectric film is directed to an operational amplifier integrator. The input to this circuit is a virtual ground so the film is held in a zero field state (to within a few millivolts). Charge is collected on a polypropylene internal reference capacitor which provides good temperature stability and low dielectric absorption. Since the op amp integrator circuit is in an inverting configuration, a second op amp is used to invert the output voltage from the first op amp. The output from the device is then reported as a voltage which is proportional to the amount of charge collected on the reference capacitor.

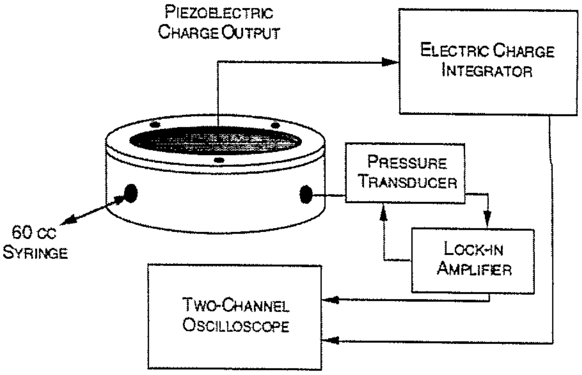

Fig. 1. Experimental setup.

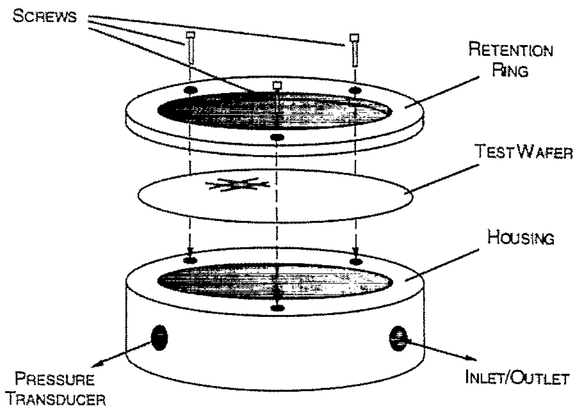

Fig. 2. Uniform pressure rig.

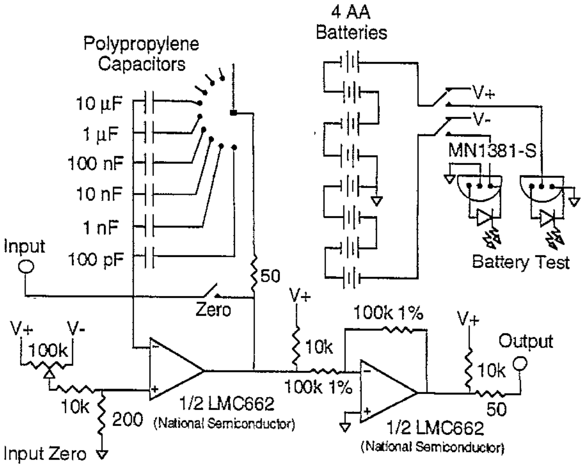

Fig. 3. Electric charge integrator.

3.3. Peripheral electronics

The pressure transducer used in our experiments is constructed in a Wheatstone bridge configuration where the output voltage across the bridge is proportional to the change in pressure within the rig housing. Electromagnetic noise was minimized by using a lock-in technique to both excite the bridge and monitor the change in the output signal. Signals from both the lock-in amplifier and the charge integrator were input to a Hewlett-Packard 54600 oscilloscope and the two traces produced were used to calculate (1) the piezoelectric charge created and (2) the change in mechanical load.

4. Stress analysis and the calculation of d_{31}

The determination of the film's transverse piezoelectric coefficient requires knowledge of the mechanical stress applied to the film. Small deflection plate theory was used together with the pressure applied to the wafer, the support radius, the substrate thickness, and the test capacitor location to calculate the principle stresses at a specific location on the silicon substrate. The bending stresses which result from a uniform pressure on a clamped circular plate are [12]:

$$\sigma_r=\frac{3p_0z}{4t^3}[(1+\nu)a^2-(3+\nu)r^2] \tag{2}$$

$$\sigma_t=\frac{3p_0z}{4t^3}[(1+\nu)a^2-(1+3\nu)r^2] \tag{3}$$

where σ_r and σ_t are the radial and tangential stresses on the plate, z is the distance from the neutral axis, t is the plate thickness, ν is Poisson's ratio, a is the support radius, and r is the distance from the center of the plate.

Because the mechanical properties of the silicon substrate and the piezoelectric films differ, wafer stresses calculated from plate theory must be corrected to determine film stress. The assumption was made that all strain applied to the substrate was transferred to the film. Knowing the stresses applied to the substrate (Eqs. (2) and (3)), the strains are calculated via generalized Hooke's law as:

$$\varepsilon_1^{\mathrm{Si}}=\frac{\sigma_1^{\mathrm{Si}}}{E_{\mathrm{Si}}}-\nu_{\mathrm{Si}}\frac{\sigma_2^{\mathrm{Si}}}{E_{\mathrm{Si}}} \tag{4}$$

$$\varepsilon_2^{\mathrm{Si}}=\frac{\sigma_2^{\mathrm{Si}}}{E_{\mathrm{Si}}}-\nu_{\mathrm{Si}}\frac{\sigma_1^{\mathrm{Si}}}{E_{\mathrm{Si}}} \tag{5}$$

where σ_1^{Si} is applied stress in the 1 direction (1 and 2 are the principal in-plane directions corresponding to the tangential or radial orientation), $\varepsilon_1^{\mathrm{Si}}$ is the strain in the 1 direction, ν_{Si} is the Poisson's ratio of the silicon, and E_{Si} is Young's modulus of the silicon. Because of the conservation of strain, Hooke's law may be written in a similar manner for the PZT film where:

$$\varepsilon_1^{\mathrm{PZT}}=\frac{\sigma_1^{\mathrm{PZT}}}{E_{\mathrm{PZT}}}-\nu_{\mathrm{PZT}}\frac{\sigma_2^{\mathrm{PZT}}}{E_{\mathrm{PZT}}} \tag{6}$$

$$\varepsilon_2^{\mathrm{PZT}}=\frac{\sigma_2^{\mathrm{PZT}}}{E_{\mathrm{PZT}}}-\nu_{\mathrm{PZT}}\frac{\sigma_1^{\mathrm{PZT}}}{E_{\mathrm{PZT}}} \tag{7}$$

The equations for σ_1^{PZT} and σ_2^{PZT} can be derived explicitly by setting $\varepsilon_1^{\mathrm{PZT}}=\varepsilon_1^{\mathrm{Si}}$ and $\varepsilon_2^{\mathrm{PZT}}=\varepsilon_2^{\mathrm{Si}}$ which yields

$$\sigma_1^{\mathrm{PZT}}=\varepsilon_1^{\mathrm{Si}}E_{\mathrm{PZT}}+\nu_{\mathrm{PZT}}\sigma_2^{\mathrm{PZT}} \tag{8}$$

and

$$\sigma_2^{\mathrm{PZT}}=\frac{E_{\mathrm{PZT}}}{(1-\nu_{\mathrm{PZT}}^2)}(\varepsilon_2^{\mathrm{Si}}+\nu_{\mathrm{PZT}}\varepsilon_1^{\mathrm{Si}}) \tag{9}$$

The expansion of Eq. (9) yields a more fundamental form of the equation written in terms of the elastic properties of the silicon and the elastic properties of the PZT film where:

$$\sigma_2^{\mathrm{PZT}}=\frac{E_{\mathrm{PZT}}}{(1-\nu_{\mathrm{PZT}}^2)}\left[\frac{\sigma_1^{\mathrm{Si}}}{E_{\mathrm{Si}}}(\nu_{\mathrm{PZT}}-\nu_{\mathrm{Si}})+\frac{\sigma_2^{\mathrm{Si}}}{E_{\mathrm{Si}}}(1-\nu_{\mathrm{PZT}}\nu_{\mathrm{Si}})\right] \tag{10}$$

The expressions derived are appropriate for small deflections of a coated wafer if the ratio of the PZT film thickness to the silicon substrate thickness is small. For the case of a PZT film on a silicon substrate, the thickness ratios are much less than 1% and deformation of the composite wafer will be governed by the elastic properties of the silicon substrate. Furthermore, because small deflection plate theory is used, the maximum deflection of the coated wafer may not exceed 20% of the thickness of the plate [12,13]. For deflections beyond that point, membrane (i.e., stretching) stresses are no longer negligible and use of the small deflection equations would result in significant error.

5. Numerical considerations

5.1. Elastic modulus of PZT Film

Eqs. (9) and (10) show that the stress on the film is dependent upon both the elastic properties of the silicon and the PZT. Inaccuracies in those quantities will carry through the stress analysis and to the subsequent d_{31} calculation. It is important to note that although the mechanical properties of silicon are well characterized [14,15] the properties of thin film PZT are not. That observation is important because at present all methods (converse [3], direct [10], or values derived from low-field measurements [16]) for the determination of the transverse piezoelectric coefficient require explicit knowledge of the material's elastic moduli. Published values have ranged from 37 [17] to 400 GPa [2] and it should therefore be noted that the disparities among d_{31} values reported in the literature could result from the different elastic moduli used in their calculation. The Young's modulus used in this investigation was taken as 101 GPa [18] and was selected in order to yield a lower limit for the d_{31} coefficient (i.e., calculated stress increases and d_{31} decreases).

5.2. Anisotropy of the silicon substrate

The anisotropy of the silicon substrate can complicate the calculation of applied stress, however, strain gauge measure-

Table 1
Elastic properties used in the elastic stress analysis

Material	Young's modulus (GPa)	Poisson's ratio
{100} Silicon substrate [14]	150	0.172
PZT thin film [18]	101	0.3

ments along the ⟨100⟩ and ⟨110⟩ directions of a bare wafer showed no significant difference in the mechanical response [19]. That result suggests that the elastic properties which govern deformation of the substrate can be represented with single values of Poisson's ratio and Young's modulus. A comparison of the experimental and theoretical strains (as calculated from plate theory) gave the best agreement when the average of the maximum and minimum in-plane elastic constants (the ⟨100⟩ and ⟨110⟩ directions) were used in the calculation. For that reason the silicon was treated as an isotropic plate and the averages of both the Young's modulus and Poisson's ratio were used in the d_{31} calculations presented here. Table 1 summarizes the mechanical properties of both the silicon substrate and the PZT film.

6. Preliminary results

Initial characterization experiments were conducted with lead zirconate titanate (PZT) thin films. The films used were synthesized on 3-in. platinized silicon substrates using a conventional sol–gel procedure [20]. Solutions with 52/48 compositions (zirconium to titanium ratio) were diluted to 0.5 M concentrations and spin coated at 3000 rpm for 30 s. Individual PZT layers were pyrolyzed at 300°C and amorphous films were rapid thermal annealed at 650°C for 60 s in air. The thickness of films measured were on the order of 0.4 μm.

6.1. Strength of poling field

Table 2 shows the variation of d_{31} with the magnitude of the applied poling field for two samples at different locations on the same wafer. Results were obtained within 5 min after poling the sample for less than 1 min and suggest that above the coercive field (typically about 50–60 kV/cm for these 52/48 sol–gel films) the d_{31} value is independent of field strength.

Table 2
d_{31} coefficients as a function of poling field

Poling field (kV/cm)	d_{31} (pC/N)	
	Sample 1	Sample 2
50	−7	−5
100	−15	−13
150	−16	−14
200	−16	−15
250	na	−13

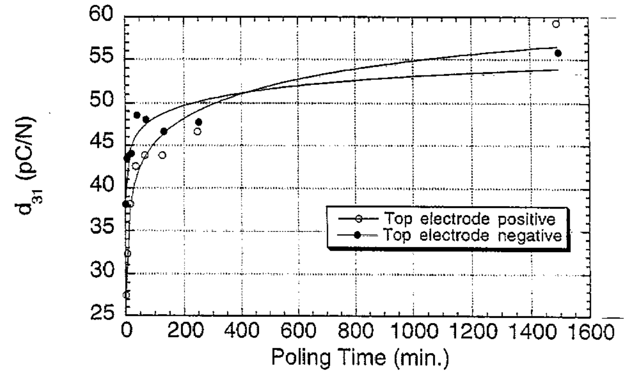

Fig. 4. The variation of d_{31} with poling time for an applied field of 150 kV/cm.

6.2. Poling time

The effect of poling time at 150 kV/cm on the measured d_{31} value of a 52/48 sol–gel PZT film is presented in Fig. 4. The data indicate a rapid increase of the piezoelectric coefficient for increased poling times from 1 to 20 min. For exposure times greater than 20 min the rate of change of d_{31} slows. The maximum value achieved was −59 pC/N for a poling time of ~21 h with the top electrode as the positive terminal. Data are also included in the plot for the same sample poled in the opposite direction. The trends reported are similar for both poling scenarios and indicate that the magnitude of the d_{31} coefficient is independent of the direction of the poling field for long poling times.

6.3. Amplitude of applied stress

The transverse coefficient was also measured as a function of the amplitude of the applied mechanical stress from ~10 to ~50 MPa (sum of the principal stresses) for a sample poled with an electric field of +150 kV/cm for less than 1 min. In a bulk ceramic, the increase of applied stress would be expected to result in the reorientation of non-180° domain walls, which would change the d_{31} coefficient from its original value. Prior experiments on the relation between stress and low-field dielectric response have indicated, however, that ferroelastic domain reorientation is negligible [8,9] in these films and, as a result, the piezoelectric coefficients should remain constant. That was in fact the case, and the results from the two experiments conducted (given in Fig. 5) serve to further illustrate: (1) the limited twin wall motion in sol–gel films, and (2) the validity of the wafer flexure technique.[1]

6.4. Variation of d_{31} over the surface of a 3 in. wafer

The d_{31} coefficient was monitored as a function of position over the surface of a coated wafer. This was done to evaluate

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[1] It should be noted that Fig. 5 is expressed as a function of the amplitude of the applied air pressure and not the biaxial stress applied to the PZT film.

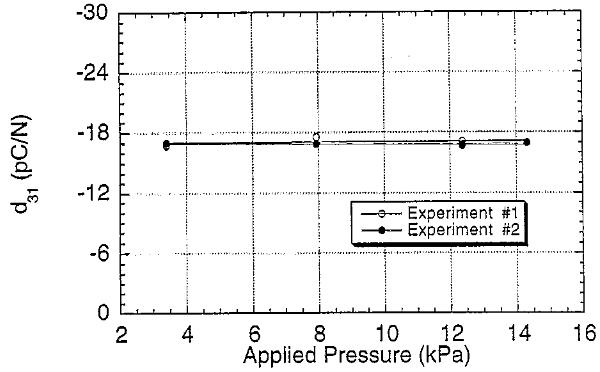

Fig. 5. d_{31} as a function of the amplitude of applied air pressure.

Table 3
d_{31} coefficients as a function of radial location on the surface of a wafer

Radius (mm)	d_{31} (pC/N)	P_r (ave.) (μC/cm^2)
2.4	−43	24
11.3	−40	21
11.3	−40	20
14.0	−44	20
16.4	−40	23
17.6	−46	21
19.5	−36	22

the consistency of the stress analysis for the wafer flexure technique. Different test capacitors on the surface of a 3 in. wafer were poled with +10 V for 10 min and allowed to stabilize for 10 min after removal of the poling field. Data collected were then correlated to the average remanent polarization (calculated from $+P_r$ and $-P_r$) for each capacitor tested. Results are given in Table 3 as a function of distance from the center of the wafer. From the data it is apparent that measured d_{31} values are independent of surface location.

7. Comments on the wafer flexure technique

The wafer flexure technique described is amenable to the rapid characterization of the transverse piezoelectric coefficient (d_{31}) of PZT thin film materials. Empirical results are in good agreement with other published values. In particular this method results in a significant reduction of cost, complexity, and the time needed to make d_{31} measurements. Furthermore, samples used are uncut silicon substrates, which eliminates the need to fashion millimeter-sized cantilever beams or diaphragms. A drawback to the uniform pressure method is its reliance on mathematical models which, to yield correct results, require explicit knowledge of the dimensions of the substrate, support housing, and test capacitor location. These factors, when coupled with the lack of data on the Young's modulus and Poisson's ratio for PZT thin films, limit the accuracy with which the transverse piezoelectric coefficient might be characterized. Additional details on calibration of this technique will be reported in a forthcoming paper [19].

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