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$e_{31,f}$ determination for PZT films using a conventional 'd_{33}' meter

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Abstract
A new and simple method is described for the determination of the piezoelectric coefficients $d_{33,f}$ and $e_{31,f}$ for piezoelectric films deposited on substrates using a conventional point-loading 'd_{33}' or 'Berlincourt' piezometer. An analytical mathematical model is developed which simulates the dynamical flexure of such films when a ring-supported sample is subject to central loading. Classical plate theory and elastic analysis are used to calculate the stresses in doped lead zirconate titanate (PZT) film for different radii of supporting rings, enabling both piezoelectric coefficients to be determined through a simple modification to the piezometer. The analytical model for the radial stresses has been evaluated in comparison with the results of finite element analysis and has shown a good correlation. The new measurement technique has been applied to both thick films of PZT and thin films of manganese-doped lead zirconate titanate (PMZT) on silicon substrates. The values of $d_{33,f}$ and $e_{31,f}$ obtained experimentally are found to be similar to those that have been determined by more elaborate methods.

1. Introduction

The use of piezoelectric thin films for applications in microelectronic devices and micromechanical systems (MEMS) requires explicit knowledge of material characteristics such as the electromechanical coupling factors, dielectric constants and piezoelectric coefficients. In bulk materials, the piezoelectric coefficients can be calculated via direct measurement of induced charge or through observation of resonance characteristics. This method becomes problematic for thin films, as the relationship of resonant and antiresonant frequencies to the piezoelectric and elastic properties of the films is less certain. In this paper a method will be described for the determination of the thin-film piezoelectric coefficients $d_{33,f}$ and $e_{31,f}$, which requires a simple adaptation (figure 1(a)) of the standard dynamic central point-loading d_{33} measurement apparatus, commonly referred to as a 'd_{33}' meter or 'Berlincourt' piezometer. Here the subscript f refers to the effective thin-film coefficient. This method is much simpler than most techniques currently employed such as laser interferometry [1, 2], cantilever flexure [3, 4], wafer flexure [5] and pneumatic loading techniques [6]. In general, these rely on specialized measurement and testing apparatus, and often require specific sample geometries (long bars for the cantilever method and whole circular wafers for the wafer flexure technique). The technique outlined here avoids complicated preparation and uses arbitrary sample substrate geometry. It may be used to determine the transverse piezoelectric coefficient for films having a thickness of up to around 20 μm. Experimentally determined values of $d_{33,f}$ and $e_{31,f}$ are similar to those obtained by other methods for thin films [3, 7].

2. Principle and theory

2.1. Analytical model

A typical test sample consists of a piezoelectric film with a Ti/Pt base electrode deposited on a planar silicon substrate. The substrate can be of arbitrary shape and the upper surface of the film typically has deposited on it an array of electrode dots, 1–2 mm in diameter. The sample is placed on a circular ring support with an electrode dot on the upper surface, centrally positioned, and the sample is dynamically loaded by the upper force-head of the piezometer (figure 1(b)). Bending of the substrate introduces a planar stress to the piezoelectric thin film. A surface charge is generated on both the top and bottom surfaces of the doped lead zirconate titanate (PZT) layer via the direct piezoelectric effect and includes contributions from both

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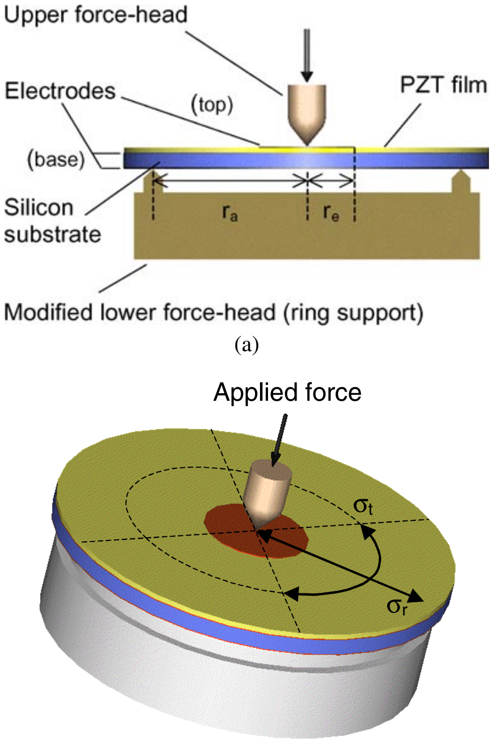


(a)

(b)

Figure 1. Schematic diagrams of (a) the test sample with a support ring and (b) the load geometry.

$d_{33,f}$ and $d_{31,f}$. By changing the geometry of the test support, the relative contributions of these two coefficients can be varied, so enabling them to be separated mathematically. The surface charge is measured by the piezometer which displays an effective piezoelectric coefficient, defined here as d' such that:

$$d' = d_{33,f} + e_{31,f}[f_1(\sigma_r) + f_2(\sigma_t)] \qquad (1)$$

where f_1 is a function of the radial stress, σ_r, f_2 is a function of the tangential stress, σ_t, and $d_{33,f}$ and $e_{31,f}$ are the longitudinal and transverse piezoelectric thin film coefficients, respectively. These functions are now derived.

The stresses in a flexed PZT-coated silicon wafer may be calculated by small deflection plate theory together with elastic stress analysis. The stresses in a simply-supported circular thin plate undergoing a small deflection from a uniform load, W, over a small central area are given by [5, 8–10]

$$\sigma_r(r) = \frac{3Wz}{4\pi t^3}\left[4(1-\nu)\ln\left(\frac{r_a}{r}\right) + (1-\nu)\left\{\frac{r_a^2 - r^2}{r_a^2}\right\}\frac{r_o'^2}{r^2}\right] \qquad (2)$$

$$\sigma_t(r) = \frac{3Wz}{4\pi t^3}\left[4(1-\nu)\ln\left(\frac{r_a}{r}\right) + (1-\nu)\left\{4 - \frac{r_o'^2}{r^2}\right\}\right] \qquad (3)$$

where σ_r and σ_t are the radial and tangential stresses in the plate, z is the distance from the neutral axis in the longitudinal direction, t is the plate thickness, ν is Poisson's ratio, r_a is the support radius, and r is the radial coordinate from the centre of the plate. The effective load radius, r_o', is given in terms of the actual load radius, r_o, and the plate thickness by [8]

$$r_o' = \begin{cases} \sqrt{1.6r_o^2 + t^2} - 0.675t & \text{if } r_o < 0.5t \\ r_o & \text{if } r_o > 0.5t. \end{cases} \qquad (4)$$

The distance z is set equal to $-t/2$ to evaluate the stresses in the top surface of the silicon wafer.

Because the mechanical properties of the silicon substrate differ from those of the piezoelectric films, the calculated stresses must be adjusted to determine the film stress. In this calculation, it is assumed that the PZT layer is completely bonded to the silicon substrate such that the strains are equal in both materials at the boundary layer between the materials and that the strain is uniform throughout the thickness of the piezoelectric film (i.e. $t_{PZT} \ll t_{Si}$). These calculations are provided in Shepard *et al* [5] and the stresses in the film can be calculated according to

$$\sigma_1^{PZT} = A\sigma_1^{Si} + B\sigma_2^{Si} \qquad (5)$$

$$\sigma_2^{PZT} = B\sigma_1^{Si} + A\sigma_2^{Si} \qquad (6)$$

$$A = \frac{E^{PZT}}{E^{Si}}\left(\frac{1 - \nu^{PZT}\nu^{Si}}{1 - \nu^{PZT\,2}}\right) \qquad (7)$$

$$B = \frac{E^{PZT}}{E^{Si}}\left(\frac{\nu^{PZT} - \nu^{Si}}{1 - \nu^{PZT\,2}}\right) \qquad (8)$$

where σ_1^{PZT} and σ_2^{PZT} are the stresses in the PZT film in directions 1 and 2—equivalent to the radial and tangential stresses at the top surface of the silicon as derived from equations (2) and (3). Note that these stresses do not exhibit cylindrical symmetry about the z-axis (normal to the plane of the film) due to the elastic anisotropy of the silicon wafer.

The piezoelectric coefficient $d_{31,f}$ is related to $e_{31,f}$ through the following equation [7]

$$e_{31,f} = \frac{d_{31,f}}{s_{11}^E + s_{12}^E} \qquad (9)$$

where s_{11}^E and s_{12}^E are the relevant elastic compliances of the PZT film at constant electric field. Considering equations (1) and (5)–(9) in conjunction with the constitutive equations for piezoelectricity [11] the following equation can be derived for the charge Q_3 produced by a load W applied by the top probe of the piezometer:

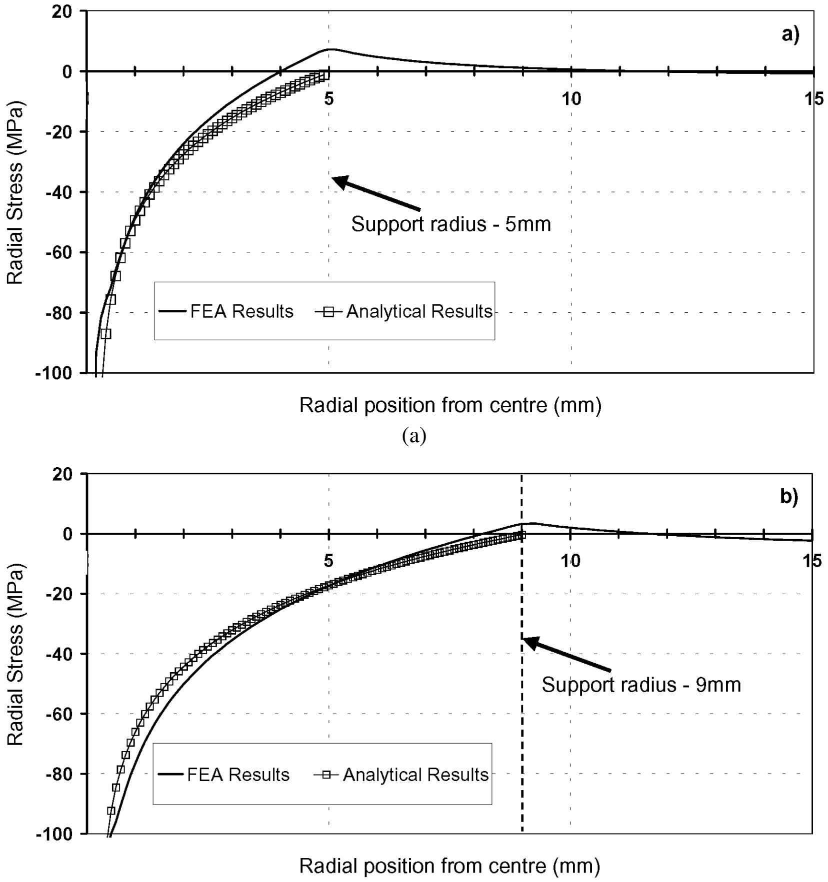
$$Q_3 = d_{33}W + e_{31}\left(\frac{1-\nu_{Si}}{E_{Si}}\right)\int_0^{2\pi} \mathrm{d}\theta \int_{r_o'}^{r_e} (\sigma_1^{Si} + \sigma_2^{Si}) r \, \mathrm{d}r \qquad (10)$$

and from this an expression can be derived for the effective piezoelectric coefficient d' measured by the piezometer:

$$d' = d_{33} + \frac{3}{t_{Si}^2} e_{31,f}\left(\frac{1-\nu_{Si}}{E_{Si}}\right)\left\{(1+\nu_{Si})\left[r_o'^2\left(\ln\frac{r_o'}{r_a} - \frac{1}{2}\right) - r_e^2\left(\ln\frac{r_e}{r_a} - \frac{1}{2}\right) + \frac{r_o'^2}{4}(1-\nu_{Si})\left(\ln\frac{r_e}{r_o'} - \frac{1}{2r_a^2}(r_e^2 - r_o'^2)\right)\right] + \frac{1}{2}(1-\nu_{Si})\left(r_e^2 - r_o'^2\left(1 + \frac{1}{2}\ln\frac{r_e}{r_o'}\right)\right)\right\}. \qquad (11)$$

Putting experimental data for d' and d_{33} into this expression permits calculation of the transverse piezoelectric coefficient $e_{31,f}$ as a function of support ring radius r_a.

The model assumes that the incident load is applied uniformly across the entire 'effective load radius'. In practice the load distribution will be influenced by, for example, the shape of the upper force-head and it may be more realistic to assume a non-uniformly distributed load symmetric about



(a)

(b)

Figure 2. Comparison of radial stress as a function of radial position in a 1 μm thin PZT film, as predicted by FEA and by mathematical analysis: (a) a 15 mm radius sample ring-supported at 5 mm and (b) ring-supported at 9 mm.

the central axis. Accordingly, the model was reassessed to determine the influence of a quadratic applied-load distribution on the wafer stresses. The change in stress distribution due to this condition was found to be very localized about the central contact and to have no significant effect on the predicted values of $e_{31,f}$.

2.2. *Finite element model*

A finite-element analysis (FEA) was carried out to test the validity of the analytical form of the stresses, as shown in equations (2) and (3). A model was set up to simulate the flexure of PZT-5H films, 1 μm thick, deposited onto a silicon substrate 525 μm thick. The model assumed a circular disc with a radius of 15 mm, simply supported on a ring at a radius of 5 mm. A uniform load of 10 N over a circular area of 0.1 mm radius was applied centrally from the top. The radial stresses calculated by FEA as a function of radial position on the film surface are shown in comparison with those determined using the analytical model in figure 2(a). The two sets are in very good agreement over the electrode radius of 0–2 mm, the range of integration required to cover the electrode area. This gives a high degree of confidence in the validity of the analytical model used for modelling the response function. Significant differences are only noticeable beyond a 2 mm radius and adjacent to the ring support. No significant variation was observed when film thickness was increased to 10 μm. The procedure was repeated using a ring radius of 9 mm (figure 2(b)). It can be inferred that for the method to retain validity, the minimum supporting ring radius used for measurement of d' should not be less than about twice the electrode radius. This condition also serves to effectively eliminate any influence on predicted results that could be attributed to a localized stress concentration in the wafer, which is observed directly below the point of contact with the upper force head.

The FEA model was extended to study the stress distributions over the surface of a stressed silicon substrate as the thickness of a PZT film was varied between 10 and 100 μm (figure 3). This revealed no significant differences in the stress distribution for PZT films up to a thickness of 20 μm, thereby putting a safe upper limit on the allowable thickness of piezoelectric material.

3. Experimental details

A Pt/Ti electrode was deposited by sputtering onto a silicon wafer (525 μm thick). The relative thicknesses of Pt/Ti/SiO$_2$ were 100/10/400 nm, respectively. Piezoelectric films were laid down by spin coating. An array of 1 and 2 mm diameter chrome–gold top electrode dots were evaporated onto the top surface of the films through a foil shadow mask to a thickness of 100 nm. A conductive electrode was also deposited on the

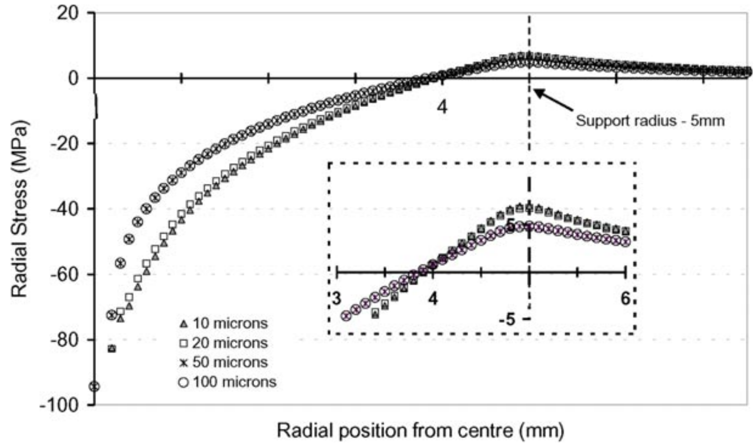


Figure 3. FEA prediction of radial stress as a function of radial position—variation in PZT film thickness.

bottom surface of the silicon substrate and electrical contact was made to the Pt/Ti using a conductive silver paint. Poling was conducted at an elevated temperature. Typically a field of 20 kV mm^{-1} was applied for 1 min at 90 °C to ensure saturation in a 1 μm manganese-doped lead zirconate titanate (PMZT) thin film. For a thick (10 μm) film of PZ26 (Ferroperm Ltd, Kvistgard, Denmark) a field of 5 kV mm^{-1} was applied for 5 min at 200 °C before cooling to room temperature under bias. The sample electrodes were shorted out overnight to eliminate space charges. The electrode spot diameter was measured on an optical microscope with a calibrated graticule and film thickness was confirmed by SEM examination of a fracture surface.

A 'Take Control'[4] piezometer was used to apply a central dynamic load to a test sample. In a simple modification, the lower 'force-head' on the piezometer was replaced with a series of circular ring supports of differing radii. The rings were manufactured from a non-magnetic stainless-steel and have narrowly tapered edges to provide the test pieces with a simple support. The upper force-head remains unchanged. An oscillating load of 0.1 N was applied at a frequency of 97 Hz, superimposed on a force bias of 10 N. The variation in the effective piezoelectric coefficient (d') of the test samples was recorded for a sequence of several different sized ring supports.

4. Results and discussion

A study was undertaken to determine the effects of a force bias as applied by the piezometer. It was found that the samples could be partially depoled by the application of an excessive bias force. It was also found that a difference in $d_{33,f}$ of around 5% resulted from over-tightening or under-tightening, by one-quarter of a turn, the screw gauge that brings the force-head into contact with the sample.

Measurements of the effective piezoelectric coefficient (d') were taken for support ring radii ranging between 2 and 9 mm. An example of the values obtained by this procedure is illustrated in figure 4(a) for a film of PMZT (1 at% Mn doped $PbZr_{0.3}Ti_{0.7}O_3$) with a thickness of 1 μm and also for a 10 μm thick film of doped PZ26. Each measurement was taken as the average of five readings and for both films the top electrode diameter was 2 mm. The value of $d_{33,f}$ was measured by the standard point load method on the piezometer (i.e. for $r_a = 0$). For the thick film of PZ26, $d_{33,f}$ was 77 pC N^{-1} and for the thin film of PMZT, $d_{33,f}$ was 46 pC N^{-1}. The effective piezoelectric coefficient was then measured using a sequence of different supporting ring radii and the value of $e_{31,f}$ was calculated using equation (11). Figure 4(b) demonstrates that although the effective piezoelectric coefficient (meter reading) increases considerably as the support ring radius increases, the calculated value of $e_{31,f}$ derived from the mathematical model remains substantially constant and this is true for all ring radii. For the thin film, $e_{31,f}$ was calculated to be 1.78 C m^{-2} and for the thick film, 4.07 C m^{-2}.

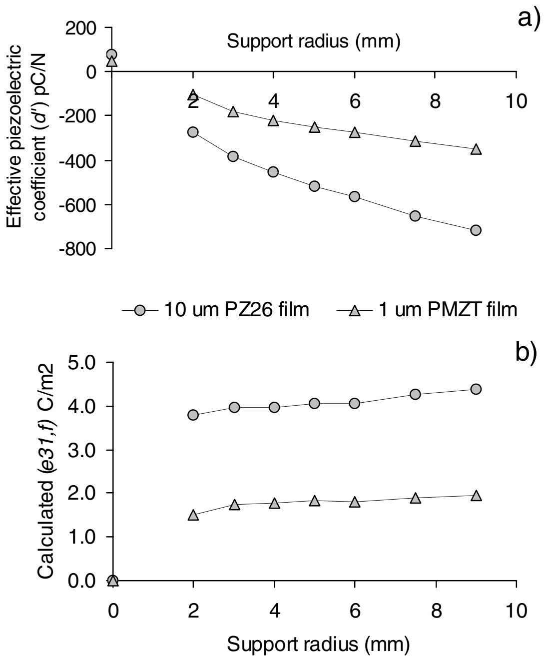


Figure 4. (a) Experimental data for sample thick and thin films and (b) calculated values of e_{31}.

The method was repeated for several poled electrode dots on the surface of a single irregularly shaped sample having a maximum surface width of 30 mm. The calculated value of $e_{31,f}$ varied by about 5% over the surface of the film and was

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[4] Take Control PiezoMeter System PM25, Take Control, Institute of Research & Development, Vincent Drive, Birmingham B15 2SQ, UK.

within experimental error. This was taken as confirmation that the method can be used confidently with irregularly shaped samples and also as an indicator of film quality. In addition, values of $d_{33,f}$ and $e_{31,f}$ can be used to accurately assess the degree of poling. The ratio of $|d_{33,f}/e_{31,f}|$ was noted as being almost constant for a particular film, independent of poling conditions.

This technique was found to be simple to use, reliable and the results are reproducible. The mathematical model assumes a perfect central alignment of the top load to the top electrode and to the support and this can be difficult to achieve experimentally. By using an average of five readings, with the sample removed and repositioned between each measurement, the errors associated with this effect were minimized.

Mathematically, the method described is only valid for flat, thin plates, where the film thickness is not more than about one-quarter that of the substrate. This condition applies because of an assumption that the PZT film has no mechanical effects on the flexure of the silicon substrate. It is also assumed that the film is thin enough for through-thickness stress variations to be ignored. The method shows excellent agreement with the results of FEA for thin films of thickness less than 20 μm deposited on a 525 μm silicon substrate. The model could be extended to thicker films by following the analysis of composite plate flexure outlined in [12].

5. Conclusion

A technique for the determination of the piezoelectric coefficients $d_{33,f}$ and $e_{31,f}$ has been outlined which requires only a simple modification to a standard 'd_{33}' meter. A mathematical model was used to predict the transverse stresses in a thin film of PZT on a silicon substrate and from this the transverse piezoelectric coefficient $e_{31,f}$ could be determined. This technique requires a simple sample preparation and goes some way towards reducing the overall cost, time and complexity of thin film measurements. Results were found to be consistent with other research carried out on thin piezoelectric films.

As a footnote, the mathematical method described here need not be restricted to a circular geometry, beam supports could equally be used, for example, and the calculations may even be somewhat simpler.

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