CANTILEVER METHOD FOR DETERMINATION OF $d_{31}$ COEFFICIENT IN THIN PIEZOELECTRIC FILMS

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Abstract: A cantilever method for characterization of thin piezoelectric films is proposed. Using the proposed cantilever method, piezoelectric coefficient $d_{31}$ of thin film piezoelectric material on various samples was determined. Cantilever based characterization method provides a fast comparison of different piezoelectric material samples, since multiple samples can be mounted simultaneously on the testing structure. It is shown how, when combined with numerical simulation, piezoelectric coefficient $d_{31}$ can be determined from fitting measured voltage response with simulated response.

Exact knowledge of geometry and material properties of cantilever and samples proved to be important in order to determine piezoelectric coefficients with sufficient accuracy. Stainless steel cantilever was adequately characterized by measuring its Young's modulus. Silicon properties are adequately determined by published data. Mechanical properties of PZT layers are on the other hand more difficult to acquire, since they are rather dependent on the actual PZT preparation procedure and composition. Nevertheless, we expect that error here introduced is small due to very thin PZT layer compared to stainless steel cantilever and silicon substrate. To improve the proposed method, based on numerical simulation results, guard chips were mounted at the side of the cantilever to reduce stress variation over samples.

Determined values of piezoelectric coefficients $d_{31}$ for PZT layers under test were in reasonable agreement with results available in the literature.

1. Introduction

When designing a new product or device, proper material selection is of basic importance. Material properties are also used in numerical analysis, when predicting device behavior. In case of piezoelectric microstructures, the properties of thin film piezoelectrics are influenced by chemical composition and other parameters of piezoelectric manufacturing process. It is thus important to have means for analyzing specific samples of piezoelectric thin films.

Due to unique properties of piezoelectric effect, piezoelectrics are important materials in micro-electromechanical system (MEMS) technology, used for actuation or sensing, energy harvesting etc. Characteristics of piezoelectrics, especially piezoelectric coefficients $d_{ij}$, play important role in device design, simulation and behavior prediction. In general, thin film materials used in microengineer-
response of different piezoelectric samples to the same mechanical stress gives immediate comparison of their basic properties such as sensitivity and linearity. Furthermore, coupling the measured results with numerical simulation based on finite element method (FEM) enables determination of absolute value for piezoelectric coefficient $d_{31}$.

The paper presents in detail the proposed technique for thin film piezoelectrics characterization and introduces a comparative method for simultaneous evaluation of multiple piezoelectric samples based on numerical simulation in combination with measured results. The result of this characterization is the absolute value of $d_{31}$ coefficient for multiple samples and comparison of piezoelectric response to mechanical stimulus. The method is practically tested on different thin film Lead Zirconate Titanate (PZT) chip samples prepared on silicon substrates. Measured results are matched with numerical simulation and piezoelectric coefficients are determined using ANSYS finite element analysis software.

2. Basic properties of piezoelectrics

Piezoelectrics are materials that respond to the applied mechanical stress with electric voltage on the electrodes. This is called the direct piezoelectric effect, which serves as a basis for sensors and generators. The effect can be reversed and it is then called converse or inverse piezoelectric effect. Here mechanical strain is induced when voltage is applied. The response is dependent on the polarity of applied voltage and can therefore vary between elongation and contraction.

Equations that describe electromechanical relations in a piezoelectric material are given in Voight notation with relations /2/:

\[
\begin{align*}
&T = [c] \{S\} - [e] \{E\} \\
&D = [e]^T \{S\} - [\hat{n}] \{E\}
\end{align*}
\]

Where $\{T\}$ is stress tensor, $\{S\}$ strain tensor, $\{E\}$ electric field vector and $\{D\}$ electric displacement vector. Material properties are described with stiffness matrix $[c]$ which includes information about Young's modulus $Y$ and Poisson ratio $\sigma$ of the material, with piezoelectric stress matrix $[e]$ (superscript T denotes matrix transpose) related to piezoelectric strain matrix $[d]$ and with permittivity matrix $[\hat{n}]$.

Piezoelectric strain coefficients $d_{ij}$ and piezoelectric stress coefficients $e_{ij}$ are related with stiffness coefficients $c_{ij}$ by matrix equation $[e] = [c]^{-1} [d]$. Piezoelectrics can be used for sensing or actuation, depending on whether the applied input load is mechanical or electrical, respectively. The two modes of operation can also be used interchangeably which makes piezoelectrics extremely versatile electromechanical materials since the same structure can act as a sensor or an actuator. Though the effect is reversible, certain considerations must be taken into account during the design of the structure /3/.

3. Piezoelectrics characterization

3.1 Bulk piezoelectrics characterization

A complete characterization process of bulk piezoelectric material includes determination of stiffness coefficients $c_{ij}$ (including Young's modulus $Y$ and Poisson ratio $\sigma$), permittivity ($\hat{n}$) and piezoelectric coefficients ($d_{ij}$).

Most widely used method adopted as IEEE standard for piezoelectric characterization is the resonance method /4/. For such characterization, piezoelectric material is prepared as a flat rectangular plate between two electrodes, forming a capacitor. The capacitor impedance $Z$ is measured at different frequencies. From $Z(f)$ diagram, the resonant ($f_r$) and anti-resonant ($f_a$) frequencies are found. Then, the elastic compliance (inverse stiffness matrix) and piezoelectric coefficients for practical purposes usually $d_{31}$ and $d_{33}$ can be derived /1/.

Direct methods for determining piezoelectric coefficients $d_{ij}$ include deformation measurements when voltage is applied to the electrodes. These methods are used to quantify the direct and converse piezoelectric effect. Direct methods are also used to investigate the behavior of the piezoelectric material in terms of hysteresis and nonlinearity, thermal behavior and aging. Mechanical deformation measurement of piezoelectric sample vs. applied voltage is used to determine piezoelectric coefficients $d_{ij}$, calculated from relation in Voight notation $S_i = d_{ij} E_j / 1$.

A different method for measuring piezoelectric coefficients $d_{ij}$ is based on direct piezoelectric effect. Here, sample is mechanically loaded, therefore the bounded electric charge becomes free, ready to flow out from the electrodes /5/. Electrodes are short circuited and electric displacement $D$ is measured. Piezoelectric coefficient $d_{ij}$ is here calculated from equation in Voight notation $D_i = d_{ij} T_j / 1$.

In order to determine the relative permittivity $\hat{n}$, capacitance measurements are carried out at low frequency, usually 1 kHz and for low AC voltage excitation levels, ranging few mV /1/. The relative dielectric constant is then calculated as

\[
\hat{n} = \frac{C t}{\hat{n}_\text{o} A}
\]

where $t$ is thickness of piezoelectric layer, $A$ electrode area, $C$ measured capacitance and $\hat{n}_\text{o}$ permittivity of free space.

3.2 Thin film piezoelectrics characterization

In general, the properties of thin film materials can differ significantly from its bulk counterparts. Therefore, adequate characterization of piezoelectric thin film properties is essential. Thin film characterization methods are usually based on similar principles as for bulk. The prevailing methods use converse piezoelectric effect where electrically excited thin piezoelectric film results in mechanical displace-
ment, which is typically in the order of a few angstroms /6/. Sometimes the direct piezoelectric effect is used. Thin film piezoelectric together with electrodes are deposited on a substrate wafer and fixed in a rigid frame above pneumatic pressure cavity /7/. Pressure in the cavity is varied thus applying different mechanical stress to the piezoelectric layer. The charge integrator is used to measure the induced charge which is used in combination with excitation pressure to determine piezoelectric coefficients $d_4$.

For determining Young’s modulus of thin piezoelectric films, several approaches exist. One of the possibilities to characterize mechanical thin film properties is presented in /8/. The experiment consists of loading a membrane with a line load applied to the middle of the span using nanoindentor. A Mireau microscope interferometer is used to observe fringes that are formed on the loaded sample. Using a CCD camera these fringes are recorded and strains determined. From known stresses and strains in the material, Young’s modulus can be determined.

### 3.3 Cantilever method for characterization of thin piezoelectric films

In this case, characterization method is focused on piezoelectric coefficient $d_{31}$ using direct piezoelectric effect.

In the proposed characterization method we introduce a cantilever with mounted piezoelectric samples on silicon substrate, with exact control of deflection. Mounting several samples simultaneously to the same cantilever provides us a comparison of piezoelectric responses of various piezoelectric materials to the same stimulus. This provides fast and accurate comparison of different piezoelectric materials appropriate for R&D work. When comparing responses of different materials, relative comparative method is usually sufficient and sometimes preferred to comparing absolute values due to its simplicity. However, determination of absolute values of piezoelectric coefficients is also possible, upgrading the proposed method with analysis of mechanical setup using appropriate numerical simulation as shown later. For this purpose, finite element analysis (FEA) software ANSYS was used.

Mechanical properties of piezoelectric and silicon were taken from literature /9,10/. Permittivity was determined from capacitance measurements.

### 4. Experimental setup

Experimental setup consisting of rectangular cross-section cantilever with mounted samples is shown in Fig. 1. Due to the simplicity of cantilever with rectangular cross-section, also analytical expressions for stress distribution exist, enabling comparison with numerical results. Proposed characterization method uses samples with thin film piezoelectric capacitor structure on silicon substrate, mounted on stainless steel cantilever. The selection of optimal samples placement is essential, usually selected for high sensitivity as the region of maximum stress distribution in the beam still having sufficient uniformity. Stress decreases in cantilever longitudinal direction towards the cantilever free end where it reaches zero. Therefore, the samples are mounted in the region of maximum stress being at the root of the cantilever. Following our simulation results, care must be taken not to induce an excessive error in the placement of samples.

![Fig. 1: Top view of the cantilever with mounted samples and side guards: (a) schematic, (b) photograph](image)

For adequate characterization of piezoelectric thin film samples, high repeatability of sample loading is essential. The testing cantilever setup, together with bonded samples represents such a test structure. Stainless steel was selected as the material for cantilever, providing possibility of high repeatable deflections. Furthermore, stainless steel cantilever is mechanically resistant and can be reused after replacing samples.

During characterization, samples are often exposed to higher mechanical stresses as during the normal sensor or actuator operation. To achieve such a wide measurement range, cold rolled austenitic stainless steel (1.4310) was selected for the cantilever realization. This material has an extended elastic range due to a special treatment during the fabrication. In this case, the cantilever returns to its initial position even after extremely large deflections.

To achieve large measured range of stresses for samples under test, the mechanical part of testing system has to provide adaptability. Therefore, 10 cm long and 18 mm wide stainless steel strips (cantilevers) of thickness 0.5 mm were cut by milling and then pressed between two rigid stainless steel plates acting as a fixed support. In this approach, the cantilever length is adjustable, resulting in increased measured range with high repeatability and accuracy.

To illustrate the characterization of piezoelectric samples with described experimental setup, various thin PZT layers
were deposited by sol-gel method on silicon chips covered by Pt/Ti as reported elsewhere /11/. Gold electrodes were placed on top of PZT layer by sputtering and shaped by shadow mask method. Thin Ti and Pt layers with thicknesses of 10 and 100 nm respectively are not significant for the overall mechanical properties of the relatively thick samples and were thus neglected in numerical simulations. As an example of the proposed characterization procedure, three samples with two different thicknesses of PZT layer were introduced, marked as samples PZT1a, PZT1b and PZT2. Due to our numerical simulations, two dummy guard chips were added at cantilever sides to achieve better stress uniformity over the samples (Fig. 1).

To assure a reliable transfer of induced mechanical stress from the cantilever to the PZT samples, a strong and stable bond between the cantilever and the samples has to be achieved. Therefore, an epoxy adhesive (UHU endfest 3000) with high bonding strength of 3000 N/cm² was used for PZT samples bonding. The extended elastic range of the selected stainless steel, in the combination with the mentioned adhesive enable highly reliable loads on testing samples, up to the silicon tensile strength. In addition, samples fixed with the selected adhesive can be easily removed at relatively low temperatures what makes the testing cantilever reusable /11/.

![Experimental setup: Taylor-Hobson traversing table and micromanipulator are used to achieve high deflection repeatability.](image)

Fig. 2: Experimental setup: Taylor-Hobson traversing table and micromanipulator are used to achieve high deflection repeatability.

To achieve highly repeatable stresses, testing cantilever with bonded samples is mounted on the fixed part of modified Taylor-Hobson 150mm Traversing Table, as shown in Fig. 2. The computer controlled worktable is motor driven in both directions, but can also be moved manually. Straightness accuracy of the worktable is within ±1 μm over the full 150mm range. In order to assure deflection repeatability, a micromanipulator with 8 mm tall pointed pin is mounted at the top of the worktable, as described in detail elsewhere /12/.

Voltage response of PZT samples is measured by Semiconductor Parameter Analyzer HP4155A, including SMU and PMU Generator Expander HP41501A.

For determination of piezoelectrics permittivity, capacitance on test capacitors is measured with HP4284A Precision LCR Meter at various frequencies, at excitation amplitude 1 V and DC bias 0 V.

5. Numerical modeling

For the purpose of simulation, commercial FEM modeling and simulation software ANSYS was used. Simulator input for cantilever test structure with samples is built using ANSYS proprietary scripting language APDL. Meshing is done using built-in automatic mesh generator. The resulting hexahedral mesh of simulated test structure is shown in Fig. 3. Local improvement of the mesh was done manually to refine mesh in structure critical regions such as thin PZT layer and to avoid badly shaped elements.

The test structure basically consists of several different layers – stainless steel (SS) cantilever, silicon (Si) substrate chip, metal and PZT layer. Electrodes and interface layers were neglected at mechanical simulation due to their small thicknesses. For modeling SS and Si materials, three-dimensional SOLID95 elements were used. PZT layer was modeled with SOLID226 elements with capability to couple mechanical and electrical quantities using piezoelectric effect.

![Generated mesh of cantilever with 3 bonded samples and two side guards.](image)

Fig. 3: Generated mesh of cantilever with 3 bonded samples and two side guards.

When we take into account material symmetry, general form of stiffness matrix \([c]\) for ceramics, permittivity matrix \([\tilde{n}]\) and piezoelectric coefficients matrix \([d]\) can be simplified /1/.

\[
[c] = \begin{bmatrix}
  c_{11} & c_{12} & c_{13} & 0 & 0 & 0 \\
  c_{12} & c_{11} & c_{13} & 0 & 0 & 0 \\
  c_{13} & c_{13} & c_{33} & 0 & 0 & 0 \\
  0 & 0 & 0 & c_{44} & 0 & 0 \\
  0 & 0 & 0 & c_{44} & 0 & 0 \\
  0 & 0 & 0 & (c_{11} - c_{13}) / 2 & 0 & 0
\end{bmatrix}
\]

(3)

\[
[\tilde{n}] = \begin{bmatrix}
  \tilde{n}_1 & 0 & 0 \\
  0 & \tilde{n}_1 & 0 \\
  0 & 0 & \tilde{n}_1
\end{bmatrix}
\]

(4)

\[
[d] = \begin{bmatrix}
  0 & 0 & 0 & d_{35} & 0 \\
  0 & 0 & 0 & d_{35} & 0 \\
  d_{31} & d_{31} & d_{33} & 0 & 0 \\
  0 & 0 & 0 & 0 & 0 \\
  0 & 0 & 0 & 0 & 0
\end{bmatrix}
\]

(5)

Due to the lack of exact information in the literature, mechanical properties of thin PZT layer were approximated by bulk values. Therefore, values \(c_{11} = 13.9 \times 10^{10} \text{ Pa},\)
The described experimental setup was used to deflect cantilever. Corresponding voltage response of the mechanically loaded PZT samples was measured with parametric analyzer as described previously. Numerical simulator was configured as discussed in the previous section, to translate the test structure into numerical model. The characterization of piezoelectric effect and related $d_{31}$ parameter was performed by fitting the measured voltage response with simulated response: $d_{31}$ parameter value was varied in the simulator until a good match between measured and calculated voltage response was found. The value of $d_{31}$ that provided best fit throughout all deflections between calculated and measured voltage response was selected as the final result for the piezoelectric coefficient $d_{31}$.

### 7. Results and Discussion

The PZT samples capacitance was measured using LCR meter at frequencies ranging from 20 Hz to 10 kHz, at excitation voltage of 10 mV. A relatively small dependence of capacitance vs. frequency was detected (Fig. 4). Measured capacitance value at 1 kHz was taken, as stated in /1/. Top electrode area was measured under the microscope. PZT layer thickness was measured after the fabrication of the layer. From data given in Table 1 the relative permittivity $\varepsilon_r$ for samples was calculated. Samples PZT1a and PZT1b are built on the same PZT layer differing only in their electrode position, regarding to the cantilever support (Fig. 1).

Simulated stress profile in PZT layer is shown in Fig. 5 (simulation path is shown in the inset). From Fig. 5 can be concluded that electrode exact position is important when performing characterization of multiple samples. Following our numerical simulations results, to minimize the difference of stress profile in neighbor samples, two longer guard chips are added at the sides, as shown in Fig. 1. Calculated stress distribution in the cantilever and samples is shown in Fig. 6a.

### 6. Procedure for determination of piezoelectric coefficient $d_{31}$

The described experimental setup was used to deflect cantilever. Corresponding voltage response of the mechanically loaded PZT samples was measured with parametric

$\begin{align*}
c_{33} &= 11.5 \times 10^{10} \text{ Pa}, \\
c_{44} &= 2.56 \times 10^{10} \text{ Pa}, \\
c_{13} &= 7.43 \times 10^{10} \text{ Pa}, \\
c_{12} &= 7.78 \times 10^{10} \text{ Pa},
\end{align*}$

were taken from literature /10/. In our case Si was modeled using anisotropic symmetric matrix with coefficients $c_{11}= 0.1657 \times 10^6 \text{ Pa}, c_{12}= 0.0639 \times 10^6 \text{ Pa}, c_{44}= 0.0796 \times 10^6 \text{ Pa}/13/.

Due to the longitudinal stress dominating in our case as confirmed by our numerical simulation, only piezoelectric coefficient $d_{31}$ was taken into account.

Boundary conditions for cantilever at FEM simulation were fixed support on the cantilever left side (deflection and its derivative equal to 0) and free deflection on the right side. To allow simple load variation, the deflection was described in the program as a parameter. Electrical ground boundary condition was set on the bottom electrode.

Standard sparse direct linear solver was used for solving the model having 85000 elements with 4 basic variables (degrees of freedom) of the problem: electric potential and displacements in $x$, $y$, $z$ direction. Sparse direct solver is a robust and fast solver for linear and nonlinear analysis, appropriate when poorly shaped elements are present in the model, such as the high aspect ratio (thickness vs. width) elements in the model of PZT layer. The sparse direct solver is based on a direct solution of equations by elimination, as opposed to iterative solvers where the solution is obtained through an iterative process that successively refines an initial guess to the final solution that is within a prescribed tolerance of the final solution. Direct elimination requires the factorization of an initial very sparse linear system of equations into a lower triangular matrix followed by forward and backward substitution. Drawback of this solver is that it requires a significant amount of memory, thus it is not suitable for larger scale models with more than a half million variables. Because sparse direct solver is based on direct elimination, poorly conditioned matrices do not pose difficulty in producing the solution /15/. Direct solver was chosen for our simulated approach since it does not exceed the recommended number of equations and there was enough computer memory available to perform computation.

Simulations were performed on Intel Core Duo 6600 64-bit processor architecture with 4GB RAM memory, running at 2.4GHz. A single simulation run with chosen solver required typically 6 minutes.

Due to the small thickness of PZT compared to the cantilever and Si substrate, the error introduced is negligible.

SS material is usually considered isotropic. The Young’s modulus of SS material was measured using nanoindentation method /13/. The measured value of SS Young’s modulus is $Y = 167.56 \text{ GPa}$.

Silicon is very well known material. Due to Si crystal symmetry, it is described by 3 stiffness coefficients $c_{11}$, $c_{12}$ and $c_{44}$. In our case Si was modeled using anisotropic symmetric matrix with coefficients $c_{11} = 0.1657 \times 10^6 \text{ Pa}, c_{12} = 0.0639 \times 10^6 \text{ Pa}, c_{44} = 0.0796 \times 10^6 \text{ Pa}/14/.

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The effect of guard chips is quantified in Fig. 6 and Table 2. As shown, the absolute stress in samples is decreased when guard chips are present. However, stress uniformity over the samples improves significantly. The relative difference in stress in both cases, without and with guard chips, was calculated between central and side samples. Guard chips thus provide more homogenous stress conditions on all samples.

According to the piezoelectric effect, voltage response is proportional to the stress, what is described by piezoelectric coefficients. Calculated voltage response of PZT samples due to calculated stress is given in Fig. 6b.

Measured time dependent voltage response of PZT samples during testing is shown in Fig. 7. Here, the cantilever was deflected to predefined values using the micromanipulator as previously described. At start, the cantilever was first manually deflected over the desired deflection value, and then after this it was released to rest in final position determined by micromanipulator. Similar procedure was applied also during the end of testing. Consequently, voltage spikes always occurred at the start and at the end of loading.

As also seen in Fig. 7, the response for constantly deflected cantilever slowly decreases with time, probably due to piezoelectric internal effects such as leakage and recombinations, and due to external effects such as input impedance of HP4155A connected to the sample. Therefore, measurement of the response was done after the spike settled down, typically after 10 seconds.

### Table 1: Measured sample parameters and calculated relative permittivity of PZT layers

<table>
<thead>
<tr>
<th>Sample</th>
<th>PZT Thickness [nm]</th>
<th>Electrodes [mm²]</th>
<th>Capacitance [nF]</th>
<th>Rel. permittivity $\varepsilon_r$</th>
</tr>
</thead>
<tbody>
<tr>
<td>PZT1a</td>
<td>740</td>
<td>0.87</td>
<td>7.67</td>
<td>737</td>
</tr>
<tr>
<td>PZT1b</td>
<td>740</td>
<td>0.87</td>
<td>7.67</td>
<td>737</td>
</tr>
<tr>
<td>PZT2</td>
<td>1554</td>
<td>0.87</td>
<td>2.40</td>
<td>484</td>
</tr>
</tbody>
</table>

### Table 2: Improvement of the stress uniformity over samples when guard chips are used.

<table>
<thead>
<tr>
<th>Stress in samples without guard chips [MPa]</th>
<th>Stress in samples with guard chips [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Position B</td>
<td>Positions A, C</td>
</tr>
<tr>
<td>13.19</td>
<td>14.74</td>
</tr>
</tbody>
</table>
Fig. 7: Measured voltage response vs. time during testing.

Measured voltage response results are graphically displayed in Fig. 8. The response amplitude is dependent on electrode distance from the cantilever support and is in correlation with simulated stress profile in PZT layer shown in Fig. 5. The voltage on PZT1a is thus considerably higher than voltage on PZT1b. PZT2 that differs in thickness and material properties produces response that is slightly higher than with PZT1a.

Determination of piezoelectric coefficient $d_{31}$ was done by using numerical simulation as described previously. Successive simulations were performed for various values of coefficient $d_{31}$ until close agreement between simulated and measured voltage response was obtained. Measured and simulated responses at various deflections for best values of piezoelectric coefficient $d_{31}$ are given in Table 3. The summary of measured values for relative permittivity $\varepsilon_r$ and piezoelectric coefficient $d_{31}$ for PZT materials under test is given in Table 4. Results obtained are in reasonable agreement with available values from the literature /7/.

8. Conclusion

Using the proposed cantilever method, piezoelectric coefficients $d_{31}$ for various thin film piezoelectrics were determined. Cantilever based characterization method provides a fast comparison of different piezoelectric material samples, since multiple samples can be mounted simultaneously on the testing structure. Furthermore, when combining experimental data with numerical simulation, piezoelectric coefficient $d_{31}$ can be determined by matching simulated results with voltage response measurements.

Exact knowledge of geometry and material properties of cantilever and samples proved to be important in order to measure piezoelectric coefficients with sufficient accuracy. Stainless steel cantilever was adequately characterized by measuring its Young’s modulus. Silicon properties are adequately determined by published data in the literature. Mechanical properties of PZT layers are on the other hand more difficult to acquire, since they are rather dependent on the actual PZT preparation procedure and composition. Nevertheless, we expect that error here introduced
is small due to very thin PZT layer compared to stainless steel cantilever and silicon substrate. To improve the presented method, based on numerical simulation results guard chips were mounted at the side of the cantilever to reduce stress variation over the samples. Determined values of piezoelectric coefficients $d_{31}$ for PZT layers under test were in reasonable agreement with results available in the literature.

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