Failure of PZT Thin Films and Membranes in MEMS

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ABSTRACT

Piezoelectric thin films used in MEMS structures, particularly diaphragms, can be subjected to strains of greater than 0.1%. To assess the fracture behavior of thin films of piezoelectric materials in MEMS, an experimental study has been carried out to determine the effects of film thicknesses, residual stresses, and electrode materials on the resulting strain at failure in biaxial tension. A bulge-testing device in combination with a laser vibrometer has been developed to assess both the residual stress and strain at failure. PZT membranes with silicon and silicon oxide support structures and gold and platinum electrode materials fail at biaxial strains between 0.14 and 0.3%. There is no clear effect of film thickness for the range of PZT films between 1 and 3 μm thick on the imposed strain to failure. Replacement of a bottom platinum electrode with a gold electrode does not alter the strain at failure of these materials. Contrary to a previous study, a reduction in the effective tensile residual stress in the diaphragm did improve the strain to failure in these structures, but this is likely due to the mode of stress balancing used in these MEMS structures. An increase in the thickness of the passivating silicon oxide in the structure (which provides a compressive stress) appears to decrease the strain to failure from 0.19 to 0.14% in these structures.

Introduction

Piezoelectric films have applications in MEMS for power generation and sensing. For power generation, micro-scale concepts to generate electrical power are fuel cells, static heat engines, and dynamic heat engines. Recent work at Washington State University has led to the development of a micro heat engine that incorporates a thin-film piezoelectric diaphragm generator of lead zirconate titanate (PbZrxTi1-xO3, PZT) [1]. PZT is attractive for MEMS applications due to its high piezoelectric and electromechanical coupling coefficients [2], and literature provides many examples of methods to use these films in MEMS structures [3]. The structure of the thin-film piezoelectric diaphragm generator chosen for the micro engine is a simple two-dimensional sandwich structure similar to that used for pressure transducers and ultrasonic transducers [4].

Bulge testing has been used in previous studies to examine the elastic and plastic properties of thin films, as well as characterizing the residual stress in the films[5]. Bulge testing can also be used to pressurize the diaphragms to failure, providing a mechanism to generate fracture in conditions similar to those used in service for MEMS at high strains. Typical operating gauge pressures for the P3 engine [1] are 5 – 25 kPa. First, the most appropriate mechanical treatment for the piezoelectric diaphragm in the P3 engine – either as a plate or a membrane – must be decided. The explicit transition from plate to membrane behavior has only been recently studied [6,7]. Komaragiri et al. [8] makes the choice of the applicable mechanics (plate vs. membrane) most clear. As an example, for the PZT diaphragms in this study, if a low (1 MPa) residual stress is present, and a very low operating pressure (0.01 kPa) is selected to act on a 5 mm diameter, 10 μm thick silicon diaphragm the methods described in [8] suggest the structure would already be in the membrane regime. Real operating pressures (above), and initial stresses are much larger than this – and therefore these conditions Therefore, we may neglect bending effects, and the diaphragm can be treated as a membrane instead of a plate.

Experimental Procedures

The PZT film chosen for this study was Pb(Zr0.4Ti0.6)O3 (PZT), where x=0.4 or 0.52. These chemistries produce films that are tetragonal (0.4) or rhombohedral (0.52) at room temperature. An acetic acid based solution of lead zirconate titanate (HoAc-PZT) was deposited onto the metallized boron doped silicon wafers in several steps [9,10,11]. The boron doped wafer is passivated with either 100 nm or 1μm of silicon oxide. The first layer is spun at 3000 rpm for 30 seconds and then placed onto a 350 ºC hotplate for 1 minute. This step is repeated for a total of three layers. The sample is then crystallized in a rapid thermal annealer (RTA) for 30 seconds at 650 ºC. This process of HoAc-PZT deposition was repeated until the desired thickness of PZT was obtained. The PZT film was patterned using standard photolithography and wet etching in a solution
that consists of approximately 0.1 vol% hydrofluoric acid, 2.5 vol% hydrochloric acid, and 97.4 vol% DI water [12]. The backside of the sample was patterned for defining membrane structures and then anisotropically wet etched to define the silicon support, resulting in membranes with an active piezoelectric layer. Membranes with lateral dimensions of between 3 and 8 mm were fabricated, while film thicknesses ranged between 0.5 and 3 μm. Both Pt and Au were used as bottom electrode structures [13].

A bulge testing system was constructed which provides the ability to pressurize membranes in both the “positive” and “negative” directions (see figure 1). The diaphragm under test was sealed to a large cavity that contains a bellows that controls the differential pressure over the diaphragm, while a laser vibrometer measured displacement or velocity. These signals, and pressure, are recorded with a computer. Membranes were pressurized to failure using a quasi-static cycling method. The samples were pressurized in the “negative” direction first, and then to the same pressure in the “positive” direction (see Figure 1), and then the pressure was cycled to a higher absolute value and tested again. This process was repeated until the membrane failed, and the final deflection at failure was recorded. Based on previous studies [15] fracture in these membranes tends to occur in the “negative” direction, which is where the PZT film is in tension via both bending at the edge of the diaphragm (which is a minimal component of the pressure – deflection relationship) and stretching.

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Figure 1. Schematic (left) of pressure applied to anisotropically etched PZT membranes (in cross section, right).

Results

Figure 2 shows the typical response of 1.5 μm thick Si support layers with 150 nm of Pt as a bottom electrode, 1 μm of PZT, and 300 nm of Au as a top electrode. As expected, the larger membranes are more compliant. The relationship between pressure and deflection can be described by

\[
p = \frac{\gamma \sigma_R t}{a^2} w + \delta \frac{E t}{a^4} w^3
\]  

(1)

where the pressure, \( p \), and center-point deflection, \( w \), of a membrane under a initial residual stress, \( \sigma_R \). The effective composite elastic modulus is defined by \( E \), and \( t \) is the full diaphragm thickness, while \( a \) is half of the side length of the laminate diaphragm, and \( \gamma \) and \( \delta \) are constants particular to the shape of the diaphragm (3.4 and 0.82 [8]) for a square membrane. The strains at any location \((x, y)\) of a pressurized membrane have a characteristic form of [9]
\[ \varepsilon = f(x, y) \frac{w^2}{a^2}, \]  

so a characteristic membrane strain, \( \varepsilon_{\text{char}} \), may be defined as

\[ \varepsilon_{\text{char}} = \frac{w^2}{a^2} \]  

\( \varepsilon_{\text{char}} \) appears frequently and is convenient for normalization. For a square membrane the strain at the center of the membrane is \( 0.883 \times \varepsilon_{\text{char}} \) for a material with a Poisson’s ratio of 0.27.

![Figure 2. Pressure – deflection relationships for various membrane side lengths. All films have 1 \( \mu \)m PZT, but the silicon and silicon oxide support structure thickness does vary between samples.](image)

By fitting experimental pressure vs. center-point deflection in the manner of Eq. 1, it is possible to determine the effective residual stress in the membrane. As noted by Nix [14], the stresses in thin films will have little impact on the underlying layers on substrates. However, in this case the use of an effective stress will allow comparisons between membranes in large amounts of tension (i.e. the 3, 4, and 6 mm membranes with 100 nm of silicon oxide), the 5 mm membranes with 1\( \mu \)m SiO\(_2\), and 8 mm membranes with 500 nm of oxide.

The pattern of PZT removed from the edges of the membrane structure was varied to determine if the lateral coverage of PZT over the membrane impacted the fracture behavior of the membranes. A typical structure, shown in figure 3, removed PZT from the high strain regions of the membrane; another pattern removed PZT from the edges of the structure (also shown in figure 3). Samples of various etching geometries were tested to failure as described in the procedures section, and the strain in the center of the membrane was recorded for each failure. In general, there was no significant relationship between the amount of PZT removed from the membrane edges and the failure strain for the high stress membranes as long as over 50% of the membrane was covered with PZT. The probability of failure was 50% at a biaxial strain of approximately 0.26%.
Bare silicon membranes (consisting of the same 1.5 μm Si thickness and a 100 nm wet thermal oxide) with 4 mm lateral dimensions were also tested using the bulge testing system. Silicon membranes failed at a mean strain of 0.31%, similar to the previous average strain at failure found by Olson [15] of 0.35%. The presence of the PZT film clearly impacts the failure of the support membrane. Several possible mechanisms are viable options for this propensity for fracture; residual stresses in the thin films, defect population in the polycrystalline PZT film, and variations in materials thicknesses (primarily in the silicon oxide layer) could be responsible for this decrease in strength in the presence of the piezoelectric structures.
To examine the effects of PZT thickness and volume, PZT films were deposited on 2.1 μm thick Si with 100 nm of thermal oxide and 175 nm of Pt as a bottom electrode, at least 5 samples were tested at each film thickness. The mean strain at failure for the various thicknesses of PZT is shown in table 1. There is no clear trend in the effects of PZT film thickness on the subsequent strain at failure. A Kruskal-Wallis statistical test was performed on these data, and suggests that there is an 85% probability that these do indeed come from different populations. Further testing will be carried out to determine if there are more significant trends. Microstructurally the films do not vary greatly in grain size or in porosity for these cases, so it is unlikely that differences could be attributed to a defect that was proportional to film thickness.

<table>
<thead>
<tr>
<th>Mean Strain at Failure (%)</th>
<th>1.0 μm PZT</th>
<th>1.5 μm PZT</th>
<th>2.0 μm PZT</th>
<th>2.9 μm PZT</th>
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<tr>
<td></td>
<td>0.16</td>
<td>0.22</td>
<td>0.15</td>
<td>0.21</td>
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Table 1. Effects of PZT film thickness on biaxial strain to failure.

A recent study has demonstrated that gold, rather than platinum, is a possible metal for use as a bottom electrode for the MEMS structures described in this study [13]. Strain to failure tests of Au and Pt bottom electrodes failed to show any statistically significant difference, suggesting that the bottom electrode has little impact on the fracture of the piezoelectric MEMS structure described in this study. A previous experiment on the effects of tensile residual stress on film fracture [14] showed that lower tensile stresses increased the strain at failure in the PZT membranes. In the current study, the tensile stresses that develop in the PZT film during processing were balanced with the addition of a compressive silicon oxide layer. Samples with oxide thicknesses of 100 nm and 1 μm, with 2 μm of PZT, were used to examine the effects of residual stress in these materials. As seen in figure 5, the strain at failure for the thinner oxides samples (high stress, approximately 60 MPa) was greater than that of the thicker oxide samples (approximately 10 MPa). As there had not been any previously observed variation in the effects of side length on the resulting strain at failure, these data suggest that the mechanism for reducing overall membrane residual stresses of using a compressive thermally grown silicon oxide could impact device performance at high strains, and merits further study.

Figure 5. A decrease in strain at failure was observed for the low stress films. The 4 mm membrane used in this case had 2 μm of PZT and 100 nm oxide, while the 5 mm membrane had 2 μm PZT and 1 μm SiO₂.
Conclusions

MEMS structures consisting of PZT films as an active piezoelectric element on silicon / silicon oxide support structures with both gold and platinum electrodes were experimentally tested using a bulge testing system that enables both positive and negative gauge pressures to apply strain to the membrane structures. The biaxial strain at failure for a variety of PZT films ranges from 0.14 to 0.3%, which is lower than the silicon / silicon oxide membrane itself (which fails at a mean strain to failure of 0.31%). The effects of PZT film thickness and the material used for the bottom electrode on the strain to failure are minimal, but a thicker silicon oxide support layer, used to balance the tensile residual stresses in the PZT film, appears to lower the strain to failure in these structures by approximately 20%. In all cases, the membranes failure appears to be primarily a result of membrane stretching stresses, with bending stresses being a minimal contributor to the film failure. However, it is important to note that the films fail when pressurized in the "negative" gauge pressure arrangement, similar to the results found in previous studies.

Acknowledgments

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References