Chapter 1

Introduction

1.1 Background

Microelectromechanical systems (MEMS) have become an increasingly important area of technology. This is due to the premise that the efficiencies of high volume production and low unit cost achieved by the microelectronics industry over the past 40 years can be translated to devices in which mechanical and electrical components are integrated within a single silicon chip (or equivalent structure) [1]. MEMS combine mechanical and electrical function in devices at very small scales. Examples include pressure sensors, accelerometers, gyroscopes and optical devices, as well as chemical, biomedical and microfluidic applications.

A key reason for the sustained technical progress and economic growth of the microelectronics industry is the speed and confidence with which complex products can be designed without the need for extensive prototyping. Design of microelectronic devices is largely enabled by the reliability of the simulation tools available and the extremely well-characterized electronic properties of the materials being utilized and the processes with which the products are created. For MEMS to achieve their promise of low unit cost and large volume production it is important that similar design, characterization and analysis procedures be developed. Several simulation tools have been developed to address this need [2, 3] and various packages are available commercially and are particularly used in the design of highly integrated MEMS devices. However, the development of standardized characterization methods and material property databases has lagged behind that of the design and simulation tools, limiting their utility.

For these reasons, the near term development of MEMS requires advances in the areas of new material development, fabrication process advancement and the development of standard mechanical characterization techniques.

A research group in Caltech, the ferroelectric group, which consists of nine faculty members, is working on new material development and new techniques for mechanical characterization with the purpose of designing MEMS devices and actuators. The aim of this comprehensive project is to use multi-scale theory tools and selected experimental methods to develop new devices, especially new actuators by using ferroelectric materials. This is expected to supplement time-consuming device prototyping process. The work presented in this dissertation is an important and key step of this ambitious project: the electromechanical characterization of devices. This will provide validation for the multi-scale materials modeling framework and will help to increase the reliability of the actuators and devices.

1.2 MEMS actuators

Active materials such as piezoelectric and ferroelectric materials are most widely used in sensors (e.g., pressure) and have founded limited applications as MEMS actuators. They are called active or “smart” materials because they produce a response of different type to that of the input or stimuli, for example, an electrical or magnetic response to a mechanical or thermal input [4]. Actuators which produce a mechanical response to an electrical, magnetic or thermal input can be used to replace existing servo mechanisms at lower size and weight or be used in applications where traditional actuators are either too bulky, inaccurate or slow, such as active damping, ultrasonics, nano-positioning and MEMS. A number of such materials exist including piezoelectrics, magnetostrictors and shape memory alloys.

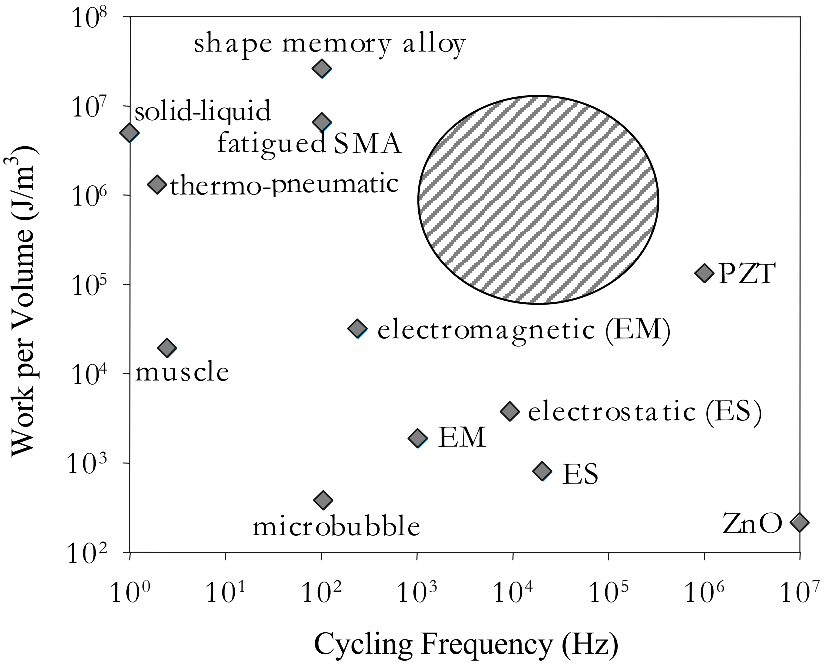

Figure 1.1. Dynamic characteristics of common microactuator systems (adapted from Krulevitch et al. [5]).

Figure 1.1, which is adapted from Krulevitch et al. [5], shows a number of actuator systems with respect to two important characteristics: work per unit volume and cycling frequency. It is observed that devices based on thermo-pneumatic systems or shape memory alloys have high work output per unit volume but lack the frequency response because of the inherent limitations in time response of the actuation system. Devices which make use of the piezoelectric (PZT) or the electrostatic effect have the desired high frequency response but with limited work per unit volume. A striking feature of the figure is that there are paucity of mechanisms or material systems that combine both high cyclic frequency and as well high work per unit volume. This points to the need and opportunity to develop novel materials and systems to fill this critical need in MEMS devices.

Piezoelectric materials, such as PZT, are ideal for applications requiring high frequency response such as ultrasonics for medical imaging, sonar, and precise displacement such as nano-positioning for fiber optic alignment or probe microscopy. These materials have the added advantage that they can be used as sensors as well as actuators allowing feedback control. The limitation of the materials is that they can only produce very small strains, up to about 0.2%. An increased actuation strain level would open a wide range of new applications that were previously impossible such as active deformable structures, miniaturized MEMS actuators, microfluidic devices and advanced biomedical applications such as robotic surgical tools.

For these reasons, there is a great need of actuators with large actuation strains. Single crystal ferroelectric materials are predicted to have large electrostriction, up to 6% for lead titatnate ($PbTiO_3$, PT) due to 90^o domain switching mechanism [6]. These materials in their thin film form could potentially result in novel MEMS devices.

1.3 Ferroelectric crystals

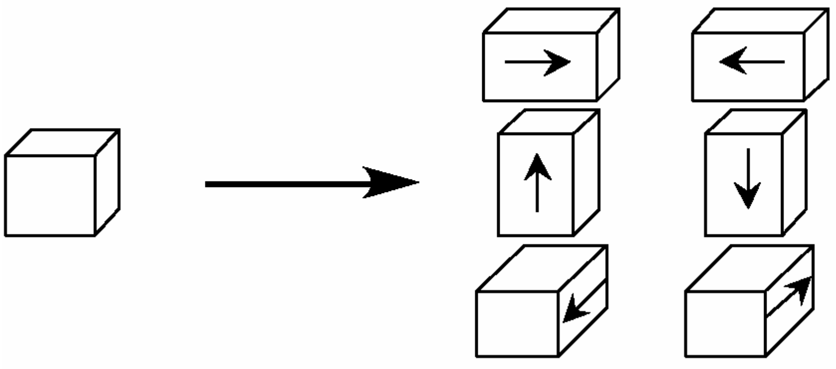

Figure 1.2. Upon cubic to tetragonal phase transition, the unit cell can take any of six equivalent combinations of strain and polarization. The arrow indicates the direction of polarization.

Ferroelectric materials have spontaneous polarization and exhibit a spontaneous, reversible electrical polarization and hysteresis behavior between polarization and electric field [4]. The ferroelectric phenomenon was first discovered in Rochelle salt ($NaKC_4H_4O_6 \bullet 4H_2O$) in 1921 [6]. Lead titanate ($PbTiO_3$) and barium titanate ($BaTiO_3$) are two common examples of ferroelectric materials. They have similar crystallographic structure (perovskite) and thus exhibit similar electro-mechanical behavior. Above Curie temperature T_c (120 ^oC for barium titanate and 490 ^oC for lead titanate) they have cubic perovskite structure and have non-polar behaviors. When cooled below the Curie temperature, they transform to a tetragonal phase. The dimensions of the unit cell are distorted along the one axis with a ratio $c/a = 1.011$ for barium titanate, resulting in spontaneous strain of 1.1%. For lead titanate, this value can be as large as 6%. In addition to the strain induced by the lattice distortion, there is a spontaneous polarization along the axis of the unit cell as indicated in figure 1.2. Because of the crystallographic symmetry, there are six equivalent orientations in space, as shown in figure 1.2. In the crystal, if a region has constant polarization orientation, that region is called a domain [4]. Figure 1.3 shows the spontaneous domain pattern in lead titanate visualized using polarization light microscopy.

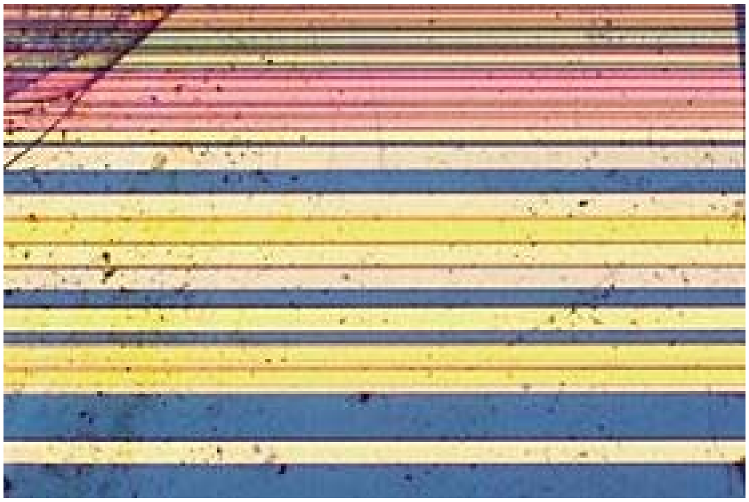

Figure 1.3. Domain pattern in lead titanate single crystal visualized using polarized light microscopy.

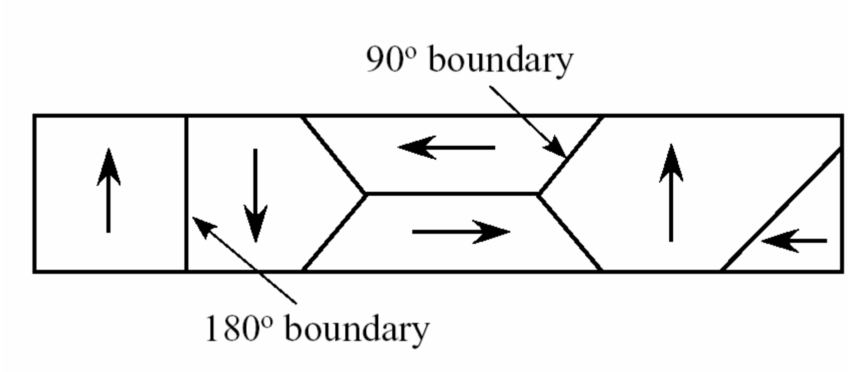

Figure 1.4. Schematic diagram of the subgranular structure of domains or regions of constant polarization separated by 90^{o} or 180^{o} boundaries.

Domains are separated by 90° or 180° domain boundaries, as shown in figure 1.4, which can be nucleated or moved by electric field or stress (the *ferroelastic* effect). The process of changing the polarization direction of a domain by nucleation and growth or wall motion is known as domain switching.

1.4 Actuator by large electrostriction ferroelectric materials

Perovskite ferroelectric crystals, such as lead titanate ($PbTiO_3$) and barium titanate ($BaTiO_3$) can have large electrostriction up to 6%, due to 90° domain switching. A recent phenomenological theory of Shu and Bhattacharya [7] described this 90° domain switching mechanism in ferroelectric materials. Motivated by this theory, experimental investigation was made to achieve large electrostriction with *in situ* observations of the domain patterns under constant compressive stress and variable electric field [8]. Figure 1.5 shows the principle of the experiments. Figure 1.6 shows the hysteresis loop of actuation strain versus electric field under 2.14 MPa constant stress [12]. A maximum cyclic actuation strain of 0.9% was achieved in this case demonstrating the feasibility of attaining large strains under combined electromechanical loading conditions.

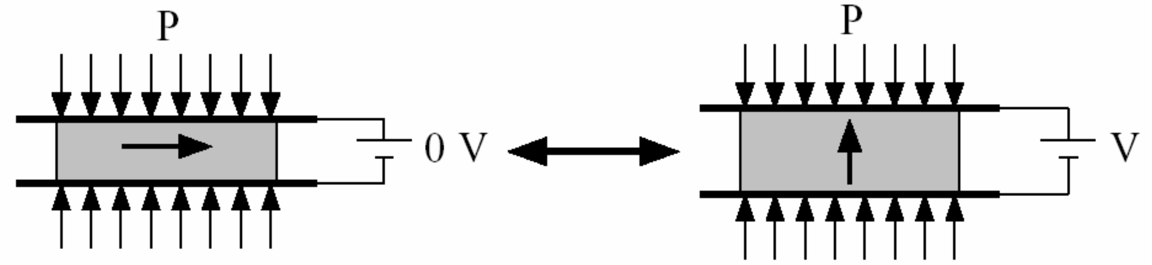

Figure 1.5. Principle of the experiments for *in situ* observations of the domain patterns under constant compressive stress and variable electric field.

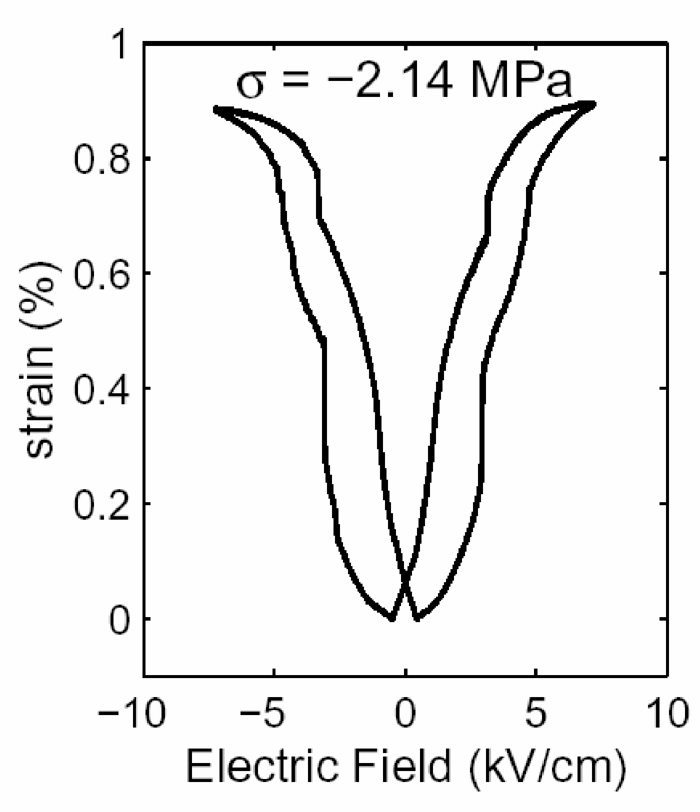

Figure 1.6. Strain vs. electric field for 2.14 MPa compressive stress [12].

Having obtained large actuation in ferroelectric bulk crystals, the Caltech ferroelectric group is working to make thin film actuators by the same mechanism. Figure 1.7 shows the schematic of a micro-pump based on ferroelectric thin film as the actuation element. Constant mechanical loading (pressure or concentrated force) is applied on the ferroelectric thin film, which has a preferred polarization orientation. According to the prediction of the theory by James and Bhattacharya [13], the thin film will form a tent shape due to the compatibility condition and 90^o domain switching in the absence of electric field. Then, with sufficient electric field applied, the reverse 90^o domain switching will take place to bring the thin film back to its original flat configuration. Such a cyclic operation could form the basis of a MEMS based micropump for potential use in microfluidic applications.

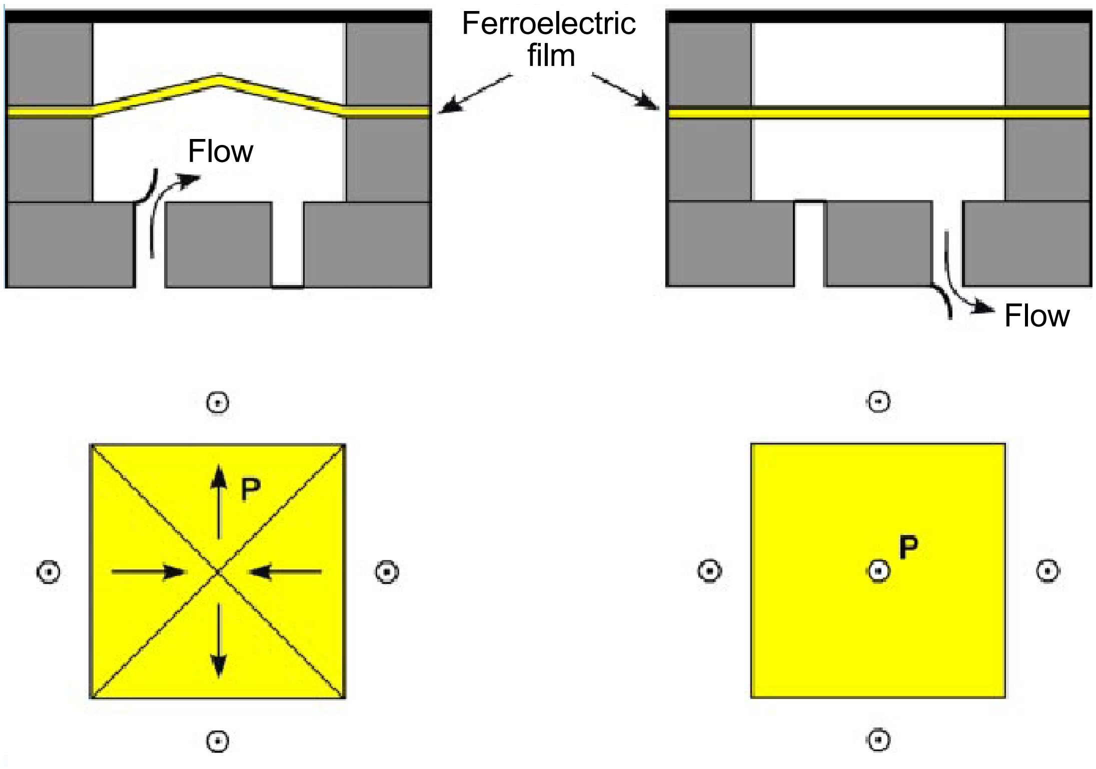

Figure 1.7. Basic principle of a micro-pump using domain switching in a ferroelectric thin film (adapted from http://www.femuri.caltech.edu).

This micropump described above is the demonstration device for the overall Caltech ferroelectric project, which includes the challenge of load control and mechanical characterization of free standing thin films.

1.5 Mechanical characterization methods of freestanding thin films

The mechanical properties of thin films have long been the focus of theoretical [9] and experimental [10] studies. As mentioned in the previous sections, mechanical characterization is essential for predicting and analyzing MEMS devices performance and reliability. However, standard methods for thin film characterization are still not available.

Many experimental techniques have been developed to determine thin film material properties such as Young's modulus, yield strength and residual stress. These techniques include micro-tension tests [11], bulge tests, and generalized indentation tests including nano-indentation [14, 15].

Micro-tensile test is a straightforward way to measure both Young's modulus and yield stress from thin films using the same general procedure employed for bulk samples but at a much smaller scale [16, 17]. However, this technique requires a potentially difficult clamping method and avoiding bending in the sample.

Generalized indentation tests using nano-indenters are suitable not only for membranes but also for shapes such as micro-bridges and micro-cantilevers. Contact mechanical load is applied on specimens by a nano-indenter, driven by the displacement of micro-actuators. The limitation of this method is that the loading and displacement range is small for many MEMS devices with large deformations.

Bulge tests applying uniform pressure on entire free-standing thin film. The displacement field is measured by interferometric techniques. By fitting the loading-deflection (*p-d*) curve, Young's modulus and the residual stress can be determined [17]. This method is suitable for membranes without defects that may result in leaks.

Beside the pressure bulge test, the above methods typically impose a fixed displacement by means of a nano-positioning motor or an on-chip actuator and measure the load. However, due to the fragile nature and the nonlinear behavior, part of the MEMS structures may be subjected to large transient load, which may cause failure under displacement control. Though these instruments may be adapted for load control by feedback loop, the response time is limited during dynamic testing and the undesired transient force problem could still exist. Moreover, for MEMS actuators made by active materials such as shape memory alloys, electrostrictive, and magnetostrictive materials, the driving force for actuation is stress rather than strain. The design of devices made from such materials requires load control and dynamic electric/magnetic/thermal loading.

For the above mentioned reasons, dynamic measurement and load control features are desired for mechanical characterization of MEMS devices and actuators. However, there is no such method available currently. New techniques need to be developed for this purpose.

1.6 Experimental techniques

In this dissertation, a new design of contact loading technique and pressure bulge test are used for mechanical characterization of thin films including that of active materials.

As mentioned before, load control is suitable for dynamic testing and can avoid undesired force on fragile materials. In this new technique described in Chapter 2, magneto-static force is used such that the force on the thin film specimen is determined directly by the distance of a pair of separated magnets. During the tests, the displacement of the sample is an order of magnitude smaller than what is required to make significant change in the magnetic force. Thus, this technique is suitable for dynamic testing and as well as quasi-static tests. Transient forces are avoided because the movable magnet is at a distance away from the sample. In order to enable dynamic experiments, the loading tip displacement which is the same as the displacement of the sample at the contact point is measured by monitoring the deflection of a single laser beam, which is reflected from a mirror attached to the end of the rigid beam. The reflected laser beam is sensed by a Position Sensitive Detector (PSD), which is used for accurate characterization of displacement of the indenter.

Bulge testing has been adapted for characterizing free standing thin films and thick films of mechanically active materials. The compact apparatus can be fitted in an X-ray diffractometer or a microscope, which supports polarized light microscopy. Combined with X-ray diffraction and polarized light microscopy, direct evidence of domain switching in single crystal thick films has been observed and the relation between the macroscopic stress and microstructure is studied.

1.7 Outline

The background and motivation for the current work is presented in the present chapter. Chapter 2 will describe a new technique for mechanical characterization of MEMS devices using contact loading method. Chapter 3 will describe the pressure bulge setup adapted for testing ferroelectric thin film. The mechanical characterization results of single layer Si_3N_4 free standing thin films and $Si_3N_4/MgO/Ba_{1-x}Pb_xTiO_3$ multi-layered thin film structure will also be presented in chapter 3. Chapter 4 will describe the microstructural evolution due to stress induced 90^o domain switching in barium titantate thick films under pressure loading and observed by combined X-ray diffraction and polarized light microscopy.. Finally, Chapter 5 summarizes the current work and makes suggestions for future work.

1.8 References

1. S. M. Spearing, Acta Mater., **48**, 179-196 (2000).
2. S. D. Senturia, Proc. I.E.E.E., **86**, 1611 (1998).
3. S. D. Senturia, Sensor Actuators A, **70**, 1-7(1998).
4. K. Uchino, *Ferroelectric Devices* (Marcel Decker, New York, 2000).
5. P. Krulevitch, A. P. Lee, P. B. Ramsey, J. C. Trevino, J. Hamilton, and M. A. Northrup, J. MEMS, **5(4)**, 270-282 (1996).
6. Y. Xu, *Ferroelectric Materials* (North-Holland, New York, 1991).
7. Y. C. Shu, and K. Bhattacharya, Phil. Mag. B, **81(12)**, 2021-2054 (2001).
8. E. Burcsu, K. Bhattacharya, G. Ravichandran, J. Mech. Phys. Solids, **52**, 823-846 (2004).
9. L. Gan, and B. Ben-Nissan, Comp. Mat. Sci., **8**, 273 (1997).
10. S. Greek, and F. Ericson, Mat. Res. Soc. Symp. Proc., **518**, 51 (1998).
11. D. T. Read, Mat. Res. Soc. Symp. Proc., **518**, 167 (1998).
12. E. Burcsu, Ph.D. thesis, California Institute of Technology, 2001.
13. R. D. James, and K. Bhattacharya, J. Mech. Phys. Solids, **47(3)**, 531-576 (1999).
14. H. D. Espinosa, B. C. Prorok, and M. Fischer, J. Mech. Phys. Solids, **51**, 47-67 (2003).
15. M. R. Begley, and T. J. Mackin, J. Mech. Phys. Solids, **52**, 2005–2023 (2004).
16. I. Chasiotis, and W. Knauss, Exp. Mech., **42(1)**, 51-57 (2002).

17. W. N. Sharpe, Jr., Mater. Res. Soc. Symp. Proc., **444**, 185–190 (1996).

18. K. Bhattacharya, and G. Ravichandran, Acta Mater., **51**, 5941-5960 (2003).

19. R. L. Edwards, and W. N. Sharpe, Exp. Mech., **44**, 49-54 (2004).

20. L. B. Freund, and S. Suresh, *Thin film materials: stress, defect formation, and surface evolution* (Cambridge University Press, New York, 2003).