



**ELECTROSTRICTIVE POLYMERS FOR  
MECHANICAL-TO-ELECTRICAL ENERGY  
HARVESTING**

THESIS

William G. Kaval IV, Captain, USAF  
AFIT-ENG-MS-17-M-038

**DEPARTMENT OF THE AIR FORCE  
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***AIR FORCE INSTITUTE OF TECHNOLOGY***

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ENERGY HARVESTING

THESIS

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## Abstract

Research of electrostrictive polymers has generated new opportunities for harvesting energy from the surrounding environment and converting it into usable electrical energy. Piezoelectric ceramic-based devices have long been used in energy harvesting for converting mechanical motion to electrical energy. Nevertheless, those materials tend to be unsuitable for low-frequency mechanical excitations such as human movement. Since organic polymers are typically softer and more flexible, the translated electrical energy output is considerably higher under the same mechanical force. Currently, investigations in using electroactive polymers for energy harvesting, and mechanical-to-electrical energy conversion, are beginning to show potential for this application. In this paper we discuss methods of energy harvesting using membrane structures and various methods used to convert it into usable energy. Since polymers are typically used in capacitive energy-harvesting designs, the uses of polymer materials with large relative permittivities have demonstrated success for mechanical to electrical energy conversion. Further investigations will be used to identify suitable micro-electro-mechanical systems (MEMs) structures given specific types of low-frequency mechanical excitations (10-100Hz).

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William G. Kaval IV

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## List of Acronyms

|                 |                                             |
|-----------------|---------------------------------------------|
| AFIT            | Air Force Institute of Technology           |
| AFRL            | Air Force Research Laboratory               |
| Al              | aluminum                                    |
| CAD             | Computer-Aided Design                       |
| CF <sub>4</sub> | carbon tetrafluoride                        |
| CO <sub>2</sub> | carbon dioxide                              |
| CTE             | Coefficients of Thermal Expansion           |
| DAQ             | Data acquisition                            |
| DIW             | deionized water                             |
| DRIE            | Deep Reactive Ion Etch                      |
| EAP             | Electroactive Polymer                       |
| EDA             | Electronic Design Automation                |
| FEM             | Finite Element Modeling                     |
| HF              | hydrofluoric acid                           |
| LPCVD           | Low Pressure Chemical Vapor Deposition      |
| MEK             | methyl ethyl ketone                         |
| MEMS            | Microelectromechanical Systems              |
| MUMPs           | Multi-User MEMS Process                     |
| N <sub>2</sub>  | nitrogen gas                                |
| O <sub>2</sub>  | oxygen                                      |
| P(VDF-TrFE)     | Poly(Vinylidene Fluoride-Trifluoroethylene) |
| PMMA            | poly(methyl methacrylate)                   |

|                                |                                       |
|--------------------------------|---------------------------------------|
| PolyMUMPs                      | Polysilicon Multi-User MEMS Process   |
| PSG                            | Phosphosilicate Glass                 |
| PZT                            | lead zirconate titanate               |
| R&D                            | Research and Development              |
| RF                             | Radio Frequency                       |
| RIE                            | Reactive Ion Etch                     |
| SCCM                           | Standard Cubic Centimeters per Minute |
| SEM                            | Scanning Electron Microscope          |
| Si                             | silicon                               |
| Si <sub>3</sub> N <sub>4</sub> | silicon nitride                       |
| SiO <sub>2</sub>               | silicon dioxide                       |
| SOI                            | silicon on insulator                  |
| Ti                             | titanium                              |
| Uv                             | Ultraviolet                           |
| VLSI                           | Very-Large-Scale Integration          |

# ELECTROSTRICTIVE POLYMERS FOR MECHANICAL-TO-ELECTRICAL ENERGY HARVESTING

## I. INTRODUCTION

### 1.1 Background

Natural energy sources are attracting a rising amount of interest due to increasing environmental concerns. Electroactive Polymer (EAP) research is one of the new opportunities for harvesting energy from the natural environment and converting it into usable electrical energy. Likewise the Microelectromechanical Systems (MEMS) field is also a rapidly advancing technological discipline, and the last several decades MEMS devices have been incorporated into a large number of technological applications. They are found in accelerometers, gyroscopes, inertial sensors, resonators, and micro-mirrors. In addition to using MEMS devices to harvest energy from vibrations, another important energy harvesting application using EAP-based MEMS devices are wearable and implantable biomedical devices, such as heart rate monitors and artificial pacemakers [7]. Simply defined, MEMS refers to miniaturized mechanical and electromechanical devices, which are constructed using microfabrication methods. MEMS structures are those devices with minimum feature sizes from several millimeters ( $mm$ ) to well below a micron ( $\mu m$ ) vary from simple structures without moving parts, to complex systems with multiple moving components [8].

## 1.2 Motivation

Piezoelectric ceramic materials, such as lead zirconate titanate (PZT), are typically used for mechanical-to-electrical energy harvesting and tend to be unsuitable for low-frequency mechanical excitations such as human movement. Polymer materials such as P(VDF-TrFE) are typically more flexible, allowing the electrical energy output to be considerably higher under the same mechanical energy input.

## 1.3 Justification

The goal of this research was to design, model, fabricate, and characterize a high fill-factor, large aperture, out-of-plane, sensor/actuation assembly capable of generating electrical charge with a future research goal of creating a system to create, collect, and store usable energy. Since the harvester performance is proportional to resonant frequency a known proof-mass can be used to establish the dominant resonant frequency. Other applications of this research were explored, such as positioning feedback for high deflection large aperture electrostatic micro-mirror assemblies currently being developed by researchers from the Air Force Research Laboratory (AFRL). These consist of a  $1mm \times 1mm$  mirror atop a  $400\mu m$  pillar. Together these structures may enable the development of broadband micro-mirror arrays capable of steering large aperture laser beams across a large field of view. Ultimately, knowledge gained from this research will eliminate the need for protruding turrets housing mechanical gimbals, thus increasing the efficiency of the optical systems affected by aircraft turbulence [9].

## 1.4 Scope

The focus of this work consists of energy harvester creation using P(VDF-TrFE) co-polymer materials. Both finite element modeling using CoventorWare<sup>®</sup> and specific changes to processing variables were evaluated and used to predict and analyze the key factors which influence the response of the end product. Further investigations into P(VDF-TrFE) processing were experimentally pursued to determine possible improvements into the device electroactive properties and performance characteristics.

## 1.5 Methodology

Membrane sensor devices and cantilever based large angle micro-mirror platform assemblies were fabricated and post-processed for harvesting energy. Initially, membrane sensor structures were fabricated in-house in order to evaluate the processing and performance of the P(VDF-TrFE) co-polymer. Commercially available PolyMUMPs prototyping services were used to fabricate the cantilever based devices. PolyMUMPs uses a predefined process and materials. Therefore, post-processing was required for deposition and patterning EAP co-polymers for energy harvesting application. As commercially available MEMS prototyping services limit the sacrificial layers to several microns the first challenge is raising the assembly high enough to allow for the large initial deflections—in excess of  $100\mu m$ —necessary to reach the desired operation angles. This problem was addressed by lifting the device platform to an initial starting position using a series of partial-bimorph cantilever beams. Upon completion samples were measured using a 3D optical surface profiler and probe station at standard testing conditions along with a National Instruments Data Acquisition unit (DAQ) in order to characterize the stimulated voltage and current response.

## 1.6 Summary

This chapter provides a brief background on MEMS technology as a basis of understanding the scale of the energy harvesting structures designed in this research. Following that, a discussion on the motivation driving the methodology and associated problems and then the justification for this approach was described. Subsequent chapters detail the theory and fabrication of the MEMS devices, the methodology for the design and characterization, results from the characterization of fabricated devices, analysis of the collected data, and recommendations for future work

## II. BACKGROUND

This chapter provides a brief description and history of concepts used or investigated in this research. The first section provides a brief introduction to MEMS to include a description of what MEMS are as well as a discussion on the basic principles of MEMS fabrication, specifically those methods used in this research. The next section covers the relevant theory related to this work such as EAP theory as well as related topics such as basic spring theory. Lastly, a summary of previous work in related areas is given.

### 2.1 Microelectromechanical System (MEMS) Fabrication

A majority of MEMS fabrication techniques used today were adopted from established microelectronic fabrication methods. Sparked by the invention of the point contact transistor in 1947, MEMS emerged during the technological revolution [10]. In 1954, C.S. Smith discovered the piezoresistive effect of semiconductor materials. This discovery showed that silicon and germanium could sense air or water pressure better than metal which led to new devices such as strain gauges, pressure sensors, and accelerometers [11]. In addition to its electrical properties, researchers began exploring new microdevices which took advantage of the mechanical properties of silicon. This innovation gave rise to microelectromechanical devices, now recognized as MEMS [12].

New MEMS technologies and applications are being developed every day. MEMS fabrication is subdivided into three processes: Microforming, Bulk Micromachining, and Surface Micromachining. Although the approaches to these processes may differ for a particular device, they all share some of the same microfabrication techniques and in some cases two or more may be used to produce one device. The following

sections provide background information on the procedures and processes common to MEMS microfabrication.

### 2.1.1 Photolithography.

Photolithography is an important processing step that is used in any MEMS fabrication. The first step of this process is to coat a wafer with photoresist, a polymer which is sensitive to Ultraviolet (Uv) light. Next, a mask, typically a chromium pattern on a quartz plate, is used to expose and pattern the photoresist. This process, shown by Figure 1, is used to pattern the features of a MEMS device for both positive and negative photoresist methods [13].

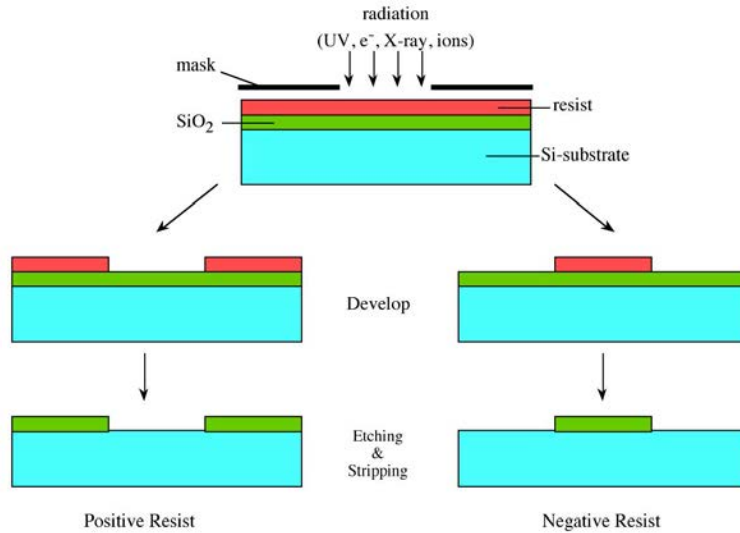


**Figure 1. Photolithography Process**  
[14]

#### 2.1.1.1 Positive Photoresist.

With positive photoresist, the Uv light causes the polymer chains to decompose. With this, exposure to the Uv light causes the chemical structure of the photoresist to become more soluble in the developer. Therefore, the mask contains an exact copy

of the pattern which is to remain on the wafer. After exposure, the developed photoresist is then washed away by the developer solution, leaving areas of unobstructed underlying material [14].

#### **2.1.1.2 Negative Photoresist.**

The negative photoresist method uses the Uv light to create cross-linking polymerization. This causes the negative resist to become more difficult to dissolve. Therefore negative resist remains on the surface wherever it is exposed as the developer solution removes only the unexposed portions. It is important to note that masks used for negative photoresist contain the inverted (or photographic negative) of the pattern to be transferred [14][13].

#### **2.1.2 Reactive Ion Etching.**

Reactive Ion Etching (RIE) is a directional etching process utilizing ion bombardment to remove material. This etch process uses a chemically reactive plasma in a vacuum chamber to aggressively etch in a vertically polarized direction. Gas enters the chamber through a control valve and is quickly evacuated by the vacuum pump. The type of gas used varies, depending on the etch process. The most common gases used for etching are carbon tetrafluoride ( $\text{CF}_4$ ) and  $\text{O}_2$  [15][16]. The RIE reaction chamber used for this research is configured so that two electrodes in the chamber create an electric field which accelerates ionized gas molecules toward the target material as shown in Figure 2. These ions have sufficient energy to physically sputter atoms out of the target material by kinetic energy transfer [13]. Horizontal etching is purposefully minimized in order to retain a high degree of anisotropy. Controlling ion density, electron temperature, and the plasma potential are among the most important parameters to consider in order to ensure uniform plasma etching.

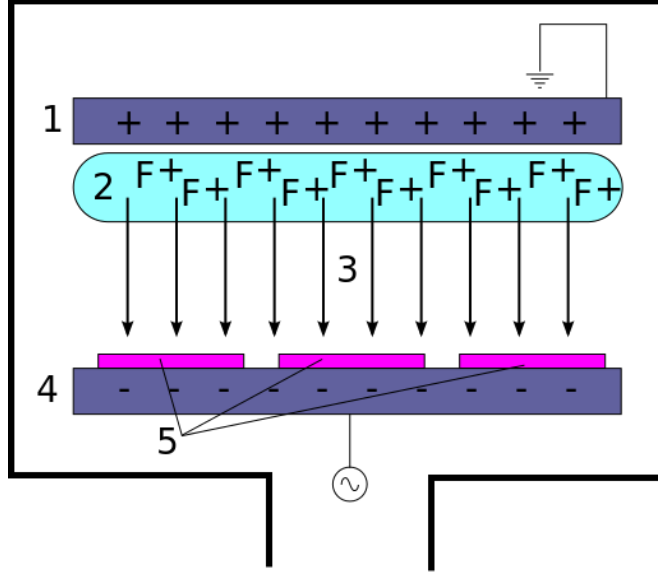


**Figure 2.** A diagram of a common RIE chamber. An RF field generates a plasma of ionized gas (2). These ions are accelerated (3) toward the samples (5) due to their attraction to the charge buildup on the sample chuck (4). Additionally, free radicals within the plasma react chemically with the material.

[13][17]

### 2.1.3 Deep Reactive Ion Etching.

Deep Reactive Ion Etching (DRIE) is a specialized method of RIE aimed at creating very deep, high aspect ratio structures. The most common specialized RIE process is the Bosch process which is used mainly for etching silicon substrates. DRIE processes operate in a similar fashion to RIE processes, however the process is tuned to be highly vertical and is capable of long, deep etches with high degrees of anisotropy up to  $90^\circ$  as seen in Figure 3. This process, developed by Robert Bosch, involves repeated cycles of shallow etches. Silicon, sulfur hexafluoride ( $\text{SF}_6$ ) is used as the active gas for the plasma etches of the silicon substrate [16]. In between these shallow etch cycles, a passivation layer is deposited on all of the exposed silicon. This polymer passivation layer results from the silicon's reaction to octafluorocyclobutane ( $\text{C}_4\text{F}_8$ ) and protects the sidewalls from the isotropic etch of the  $\text{SF}_6$  [5]. The passivation layer on the bottom of the trench is selectively removed by the vertical ion bombardment,

while leaving the sidewalls intact. Once this protective layer has been removed from the bottom of the etch, the  $\text{SF}_6$  begins to isotropically etch the freshly exposed silicon at the bottom and the entire process repeats itself over again until the desired depth has been reached [13][16].

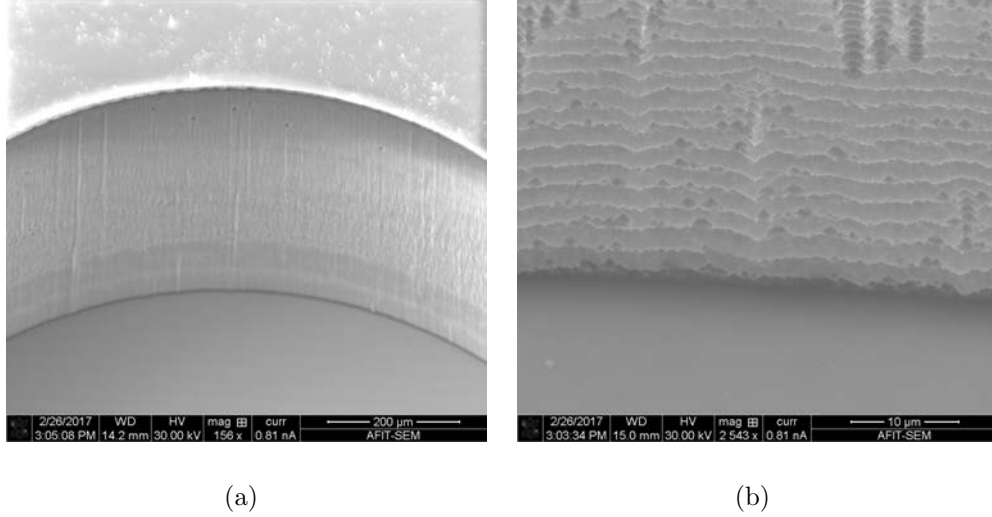


**Figure 3.** (a) SEM image of fabricated SOI membrane pillar using the SPTS Pegasus 6 deep reactive ion etch (DRIE) tool. The Pegasus 6 DRIE tool uses the Bosch process with a high power ICP source capable of fast (over  $20\mu\text{m}/\text{min}$ ), high aspect ratio (up to 100:1) anisotropic etching of silicon; (b) SEM image of scalloping on the side of the membrane pillar as a result of the iterative shallow etch cycles in the DRIE process

#### 2.1.4 Bulk Micromachining.

Bulk micromachining is a subtractive process which addresses the need for three-dimensional microstructures by removing material from the bulk substrate, typically a  $300\mu\text{m}$  to  $500\mu\text{m}$  thick silicon layer. The removal of material in this process is accomplished using physical or chemical techniques, such as dry or wet etching [18]. The remaining portion of the substrate forms the desired three-dimensional geometric structure. The key factor of bulk micromachining is the role which the crystal orientation of the substrate plays in the etching profile. Figure 4 shows a unit cell representation of the crystal structure of silicon which describes the smallest atomic configuration constructs a lattice when repeated.

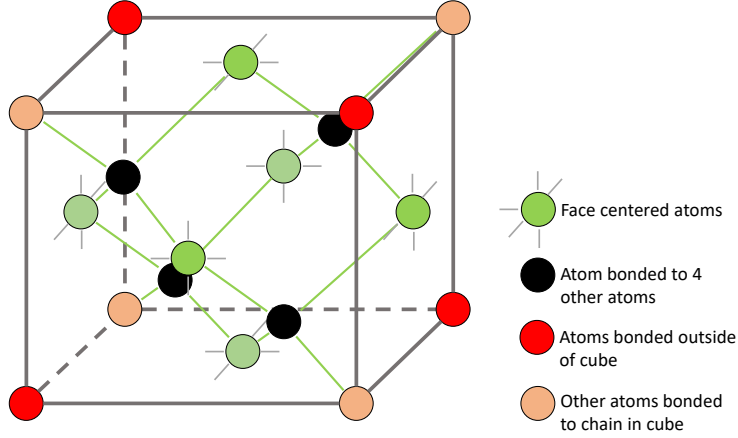


**Figure 4. Illustration of the crystal structure of silicon**

Etching can occur in two forms resulting in an isotropic (crystal orientation-independent) or an anisotropic (crystal orientation-dependent) profile. Isotropic etches remove the material evenly in all directions making it difficult to construct precise geometric structures. Anisotropic etches take advantage of the substrate material's crystalline structures to create the desired geometric structure. Figure 5 shows the crystalline structure's surface density of atoms according to the orientation of the face. The materials most common to anisotropic etch techniques are those with a

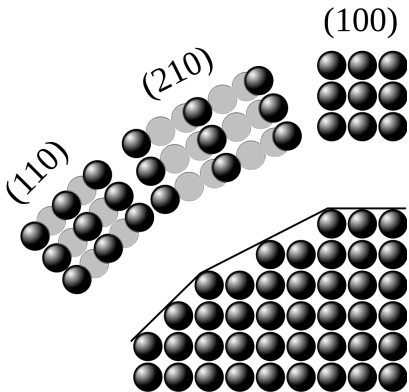


**Figure 5. Surface density of atom according to the orientation of the face**  
[19]

diamond lattice structure. The importance of this lattice structure, when etching, becomes more apparent once the individual crystal planes are observed. As seen in Figure 5, this structure is much denser in certain orientations [18][9].

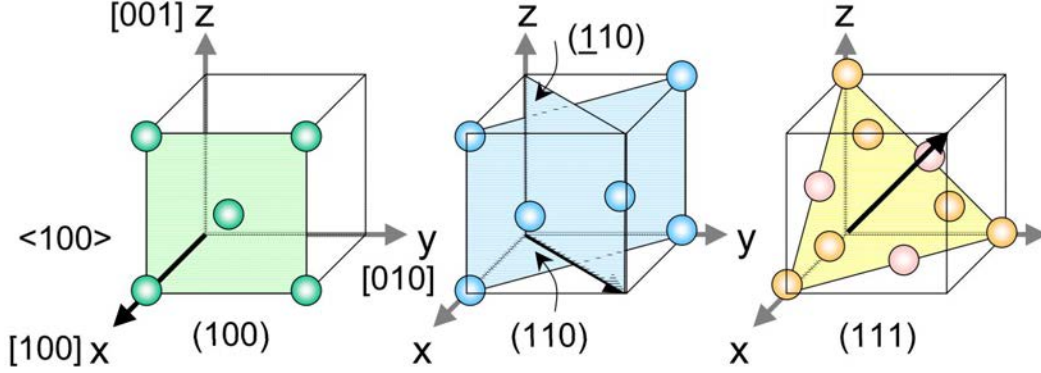


Figure 6. (From left to right) view of (100) plane showing the lattice atomic layout; view of (110) plane with respect to a unit cell; (f) top view of (111) plane with respect to a unit cell

[18]

Figure 6 shows the locations of the three primary crystal planes in a unit cell of a diamond lattice structure. When the perspective is shifted as seen in Figure 6, views of the individual crystal planes show just how much the atomic layouts vary. Atomic configurations where the atomic layout is dense tend to exhibit slow etch rates. This, coupled with a build up of higher concentrations of etched material acts as an etch stop to create unique etch profiles. The top right detail in Figure 7 shows an example of an anisotropic etch of a silicon wafer with a (100) crystalline orientation. First a layer of photoresist is deposited onto a silicon wafer and an etch window is patterned and developed using photolithography. Due to the different atomic densities of the planes, the etchant more aggressively attacks the substrate in the (100) plane versus the (111) plane. This selective etch is what makes it an anisotropic process. Figure 6(far right) shows how the (111) plane acts as an etch-stop as seen in Figure 7(bottom right). As illustrated in Figure 7(top right), due to the different atomic densities of

each of the planes, and consequently different etch rates, the (111) plane produces a unique etch profile with sidewalls at an angle of  $54.74^\circ$ .

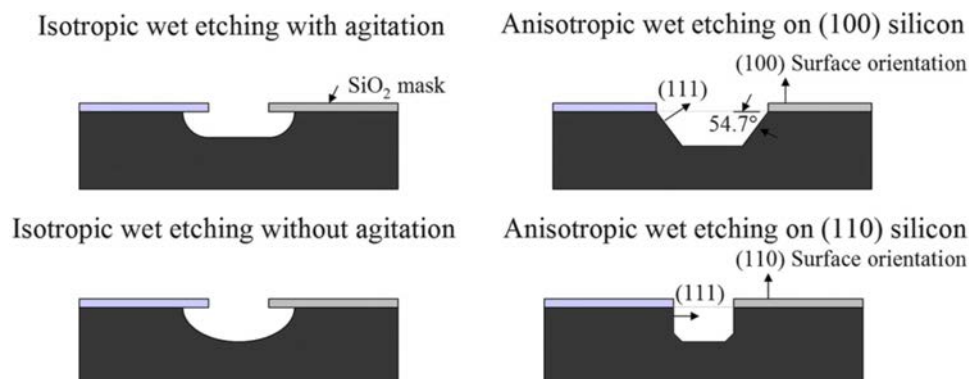


**Figure 7. Examples of both Isotropic and Anisotropic wet etching on Si**

[14]

### 2.1.5 Surface Micromachining.

Surface micromachining is the fabrication of micromechanical structures through the deposition and etching of thin structural and sacrificial films. Simple microstructures like beams or membranes as well as complex structures such as cantilevers or encapsulated resonators can be fabricated on top of a silicon substrate. Polysilicon is usually used as the structural material and silicon dioxide ( $\text{SiO}_2$ ) is typically used as the sacrificial layer. One of the main features of the surface micromachining technology is the ability to integrate MEMS and Very-Large-Scale Integration (VLSI) on the same chip. Through the use of VLSI compatible batch processing, low cost MEMS fabrication can be achieved for high volume applications [18]. Figure 8 shows the typical process how a basic cantilever beam can be produced using surface micromachining techniques. Surface micromachining typically produces one of three types of layers: insulation layers, sacrificial layers, and structural layers [11]. Insulation layers, while not shown in Figure 8, are vital to the operation of most MEMS devices; they provide a barrier between components of a microdevice, enabling electrical operation

of mechanical components. Sacrificial layers, seen in Figure 8(1-4) and also referred to as spacing layers, are used to create spacing between component layers [8]. They are constructed using a material, such as  $\text{SiO}_2$ , which can be etched away without removing other component layers of the device. Once the sacrificial layer is removed, it creates three-dimensional components and provides the spacing required to allow the freedom of movement for the mechanical components of the device. Lastly, the structural and mechanical layers are typically formed of silicon, polysilicon, metals, or alloys. As the name suggests, these are the layers which comprise the structural components of the device [9][11].

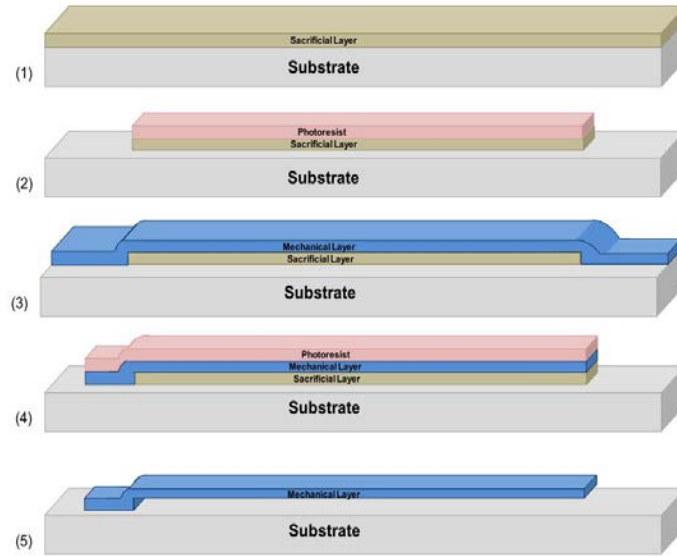


**Figure 8.** Cross-sectional view of a surface micromachining fabrication process for a cantilever beam: (1) sacrificial layer deposited onto a substrate; (2) photoresist masking layer applied, exposed, and developed and the sacrificial layer etched; (3) mechanical layer deposited; (4) photoresist masking layer applied, exposed, and developed and the mechanical layer etched; (5) sacrificial layer etched, device is now released

[9]

#### 2.1.5.1 PolyMUMPS.

PolyMUMPs is a commercial three-layer polysilicon surface micromachining process. The process is shown in Figures 9 & 10 and begins with 150mm n-type (100)

silicon wafers. The surface of the wafers are first heavily doped with phosphorus to

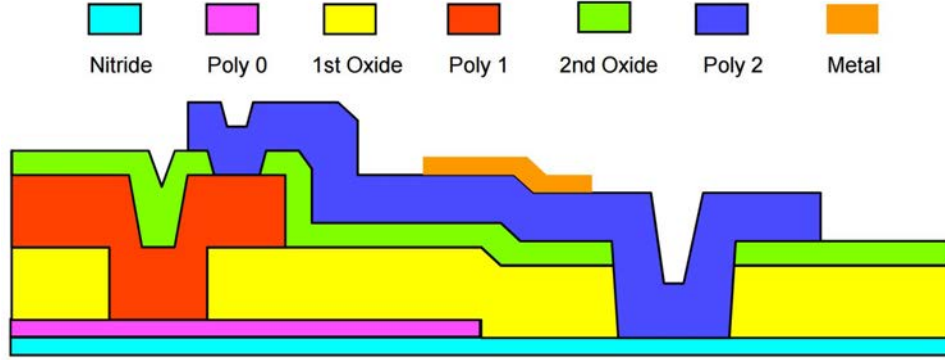


**Figure 9. Cross-sectional view of all seven layers of the PolyMUMPS process**  
[8]

reduce/prevent charge passing through to the substrate from electrostatic devices on the surface. A  $0.6\mu\text{m}$  silicon nitride ( $\text{Si}_3\text{N}_4$ ) layer is then deposited on the wafers as an electrical isolation layer. This is followed by the deposition and patterning of Poly0, a  $0.5\mu\text{m}$  Low Pressure Chemical Vapor Deposition (LPCVD) polysilicon film. A  $2.0\mu\text{m}$  Phosphosilicate Glass (PSG) sacrificial layer is then deposited by LPCVD. This layer of PSG is known as the first oxide ( $1^{\text{st}}$  oxide) and is removed at the end of the process to free the first mechanical layer of polysilicon. The next step etches anchors and dimples into the sacrificial PSG layer ( $1^{\text{st}}$  oxide) in an RIE system. The anchors are etched entirely through the sacrificial oxide layer and mechanically connect the subsequent polysilicon layer (Poly1) to the bottom Poly0 layer. The dimples are etched  $0.75\mu\text{m}$  partially through the sacrificial oxide layer which provide a small bearing surface for the mechanical polysilicon layer and avoid stiction. Following the anchor and dimple etches, a  $2.0\mu\text{m}$  thick layer of polysilicon is deposited. This polysilicon layer called Poly1 is patterned and a second sacrificial,  $0.75\mu\text{m}$  thick, oxide layer is deposited and vias are once again etched. A third and final  $1.5\mu\text{m}$  thick polysilicon layer, Poly2, is deposited and patterned. In the final deposition step the wafer is coated with photoresist where the Metal layer is lithographically patterned.

The Metal (gold with a thin adhesion layer) is deposited by lift-off patterning which does not require etching. The photoresist and unwanted metal (atop the photoresist) are then removed in a solvent bath. In the final processing step the structures are

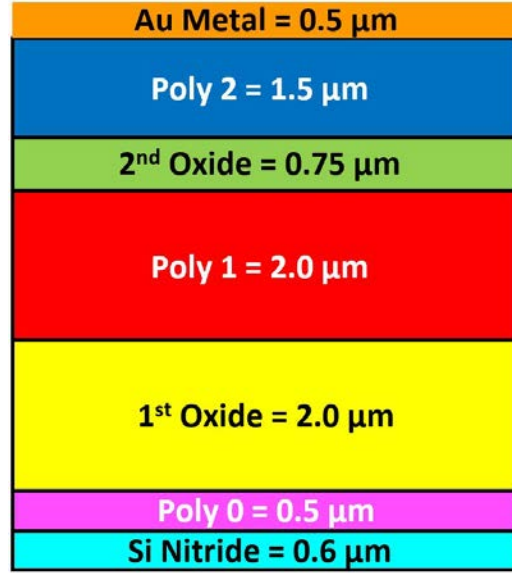


**Figure 10.** A schematic illustrating the layers and their corresponding dimensions used in the PolyMUMPs process

released by immersing the chips in a 49% HF solution. This release step removes the untrapped sacrificial oxide layers (1<sup>st</sup> Oxide and 2<sup>nd</sup> Oxide) freeing the first and second mechanical layers of polysilicon (Poly1 and Poly2) [8]. Figure 11 shows an SEM image of a micro-mirror platform assembly fabricated and released using the PolyMUMPs process. This image illustrates the ability to fabricate an electrostatically actuated micro-mirror platform assembly with this process.

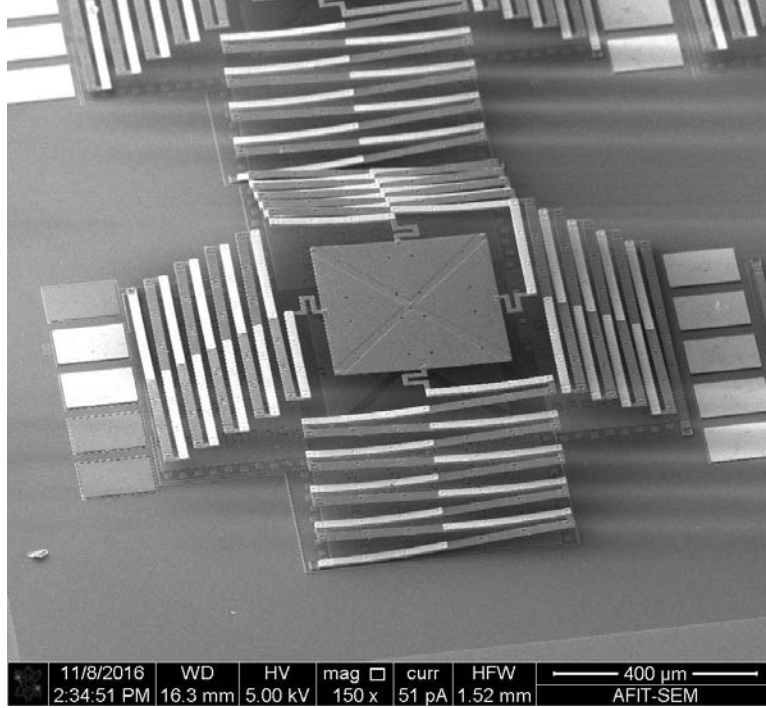


Figure 11. SEM image of a released micro-mirror platform assembly actuated by four 8-beam “zipper” actuators

### 2.1.6 Microforming.

In addition to surface micromachining and bulk micromachining, there is a third micromachining technology called LIGA, which is a German acronym for “Lithographie Galvanoformung Adformung”. Microforming addresses the need to produce high aspect ratio devices. As such, the standard microforming process used today is called the LIGA processes. This is primarily a non-silicon based technology and requires the use of synchrotron generated x-ray radiation [20]. Rather than borrowing from established microfabrication techniques, it evolved from macrofabrication techniques used in processes like electroplating and injection molding [11]. The process is shown in Figure 12 and begins by coating an electrically conductive surface with a thick layer ( $300\mu m$  to  $500\mu m$ ) of x-ray resist, poly(methyl methacrylate) (PMMA). The PMMA resist is then exposed to x-ray radiation through a mask. These x-rays break

the the polymer bonds in the PMMA. Following exposure, the exposed PMMA becomes more soluble in the developer. The exposed PMMA is then washed away by the developer solution, leaving windows of the bare underlying material. Metal is then electroplated on to the electrically conductive surface exposed during the development step. Finally, the remaining resist is removed leaving behind a high aspect ratio metal structure [20][11][6].

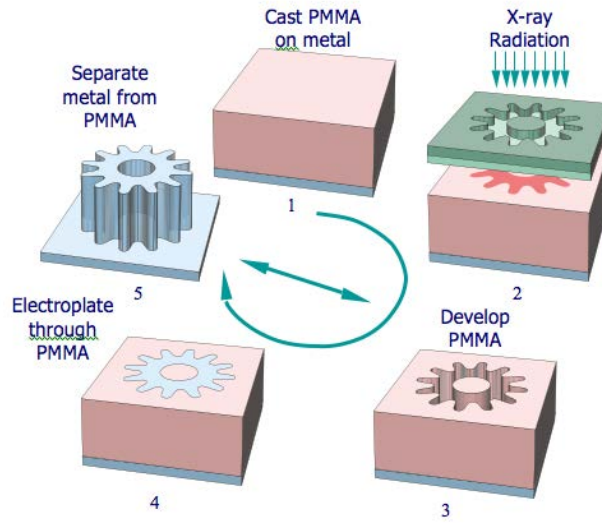


**Figure 12.** An illustration of the steps involved in the LIGA process to fabricate high aspect ratio MEMS devices

[20]

Because LIGA requires a special mask and a synchrotron (X-ray) radiation source for the exposure, the cost of this process is relatively expensive. The dimensional control of this process is quite good and the tool insert can be used many times before it is worn out [20].

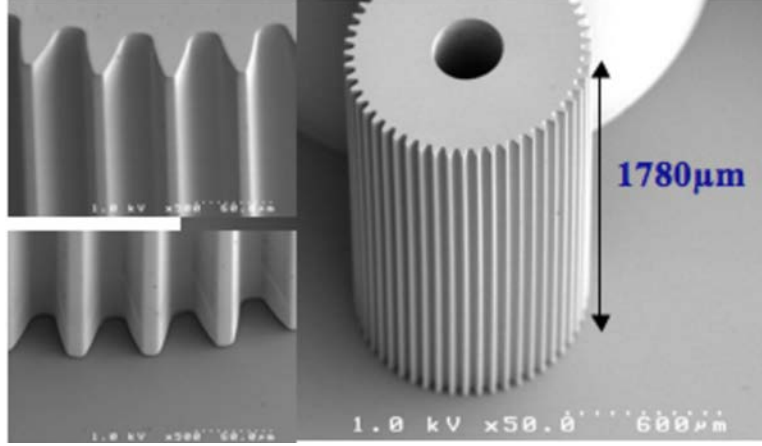


**Figure 13. A tall, high aspect ratio gear made using LIGA technology**

[20][6]

### **2.1.7 Hybrid MEMS Fabrication.**

The method silicon on insulator (SOI) is one of both integrated circuit (IC) and MEMS fabrication in which bulk silicon wafers are replaced with wafers that have three layers. These layers are a thin silicon layer ranging from a few nanometers to several microns thick, a thin underlying layer of an insulating material, and a thick support layer known as a handle [21][6]. The insulating layer is created by flowing oxygen onto a plain silicon wafer and then heating the wafer to oxidize the silicon, thereby creating a uniform buried layer of silicon dioxide.

As illustrated in Figures 14&15, a SOI wafer is used as the starting substrate with a handle (substrate) thickness of  $400 \pm 5 \mu m$ , a oxide thickness of  $0.25$  to  $2 \pm 0.05 \mu m$ , and a silicon thickness of  $10$  or  $25 \pm 1 \mu m$  [22]. First, the silicon layer is doped and can be used for MEMS structures, resistor structures, and/or electrical routing. A backside handle substrate etch allows for through-hole structures. In addition, a shadow-masked metal process allows for coarse Metal features such as bond pads, electrical routing, and optical mirror surfaces. Finally, a second pad-metal feature

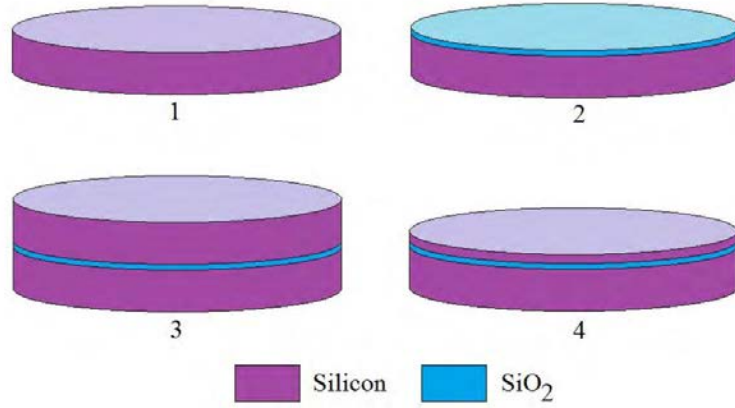


Figure 14. Step-by-step process of the SOI fabrication process. Starting with a plain silicon wafer (1), a thin oxide layer is grown to the desired thickness (2). Following the oxide growth, a second silicon wafer is bonded on top of the oxide layer at high temperature (3). Finally, the bonded silicon wafer is thinned to the desired thickness via chemical mechanical polishing (CMP).

[6]

allows finer metal features and precision alignment but limited to areas not etched in the silicon device layer [21][22].

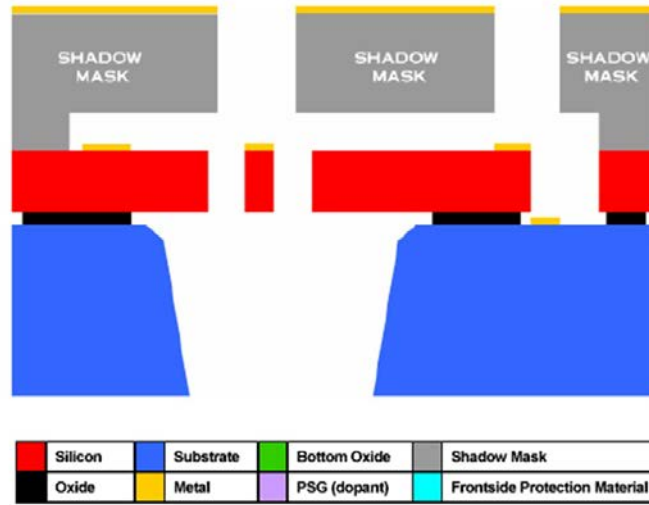


Figure 15. SOIMUMPs Process Flow

[22]

## 2.2 MEMS Transducers

A transducer is a device that converts power from one form to another for purposes of measurement or control. Most commercialized MEMS transducers are electro-mechanical where a mechanical motion, displacement, or stress accompanies the input power, whereas some type of electrical signal accompanies the output power, or vice versa [23]. Other types of transducers include thermal, chemical, optical, and biological type devices. Simply put, transducers are devices that convert one form of energy to another, and in terms of MEMS devices, it refers to both sensors and actuators [23][9].

### 2.2.1 Sensors.

Sensors are devices which measure some type of physical quantity in their environment and output a signal, usually electrical, to relay that measured parameter change [27, 28]. There are numerous types of sensors which can be divided into categories based on their domains of operation: Electrical, measuring voltage, current, or charge; thermal, which measures changes in temperature, heat, and heat flow; mechanical, which measures changes in force, pressure, velocity, acceleration, or position; chemical, which monitors changes in chemical concentration, composition, and reaction rate; magnetic, which measures magnetic field intensity, flux density, or magnetization; radiant, which measures magnetic wave intensity, wavelength, polarization, and phase [9]. Recognizing that there are nearly an infinite number of possible applications for sensors and the considerable overlap between categories, it is clear that there will be some cases where a sensor device will not fit into any one category or may belong to more than one. Generally speaking, sensors take in information from the outside world and relay a signal to the system, effectively converting mechanical energy into electrical energy [9].

### 2.2.2 Actuators.

On the other hand, actuators take a signal from the system and physically interact with the outside world (converting electrical energy into mechanical energy) [25]. Ideally, an actuator would operate using low power, be capable of fast switching speeds, maintain robustness and efficiency across all ranges of mechanical and environmental conditions, have a high power to mass ratio, and be linearly proportional between a control signal and operation parameters such as force, torque, and speed [9]. Unfortunately there is no such thing a perfect actuator. In some applications one ideal attribute contradicts another. In practice, actuator design is an exercise in trade-offs, maybe conceding actuation speed to reduce operating power or decreasing device robustness in order to increase the range of motion [9]. Much like sensors, there are a variety of actuation methods for actuators, the more commonly used methods are: thermal, using the expansion of a heated material to apply a force; thermal bimorph, which takes advantage of the difference in coefficients of thermal expansion (CTE) of two bonded materials to apply a force; electrostatic, using attractive force between two oppositely charged plates; piezoelectric, using an electrically-induced strain or stress proportional to an applied electric field to apply a force (opposite a sensor) [9].

## 2.3 Cantilever Beams

Cantilever beams are a common MEMS device used for transducers. Figure 16 shows a beam having one end fixed to a rigid structure while the other end is allowed to move freely. Typical applications of MEMS cantilever beams are: electrostatically driven relays, piezoelectric sensors, and electrothermal actuators. The basic parameters to consider when characterizing the functionality of cantilever beams are the spring constant, denoted by  $k$ , and the resonance frequency,  $f_{res}$  [24][9]. These are used in conjunction with specific material properties of the cantilever beams in order

to predict the operation characteristics of cantilever beams. Hooke's Law, Equation 1, is used to characterize the spring constant in relation to an applied force,  $F$ , and the resulting deflection of the beam,  $\delta$ .

$$F = -k\delta \quad (1)$$

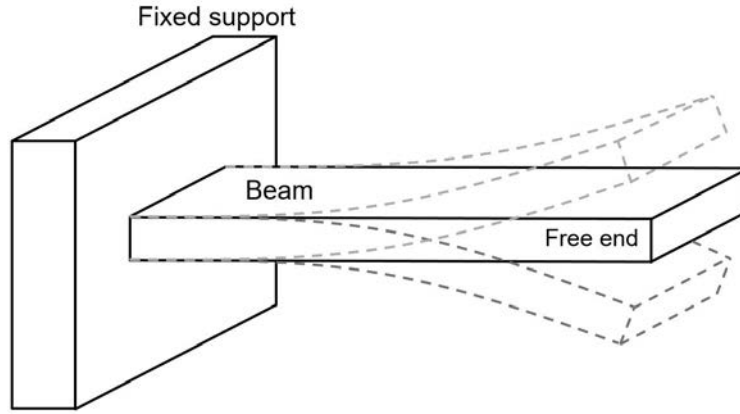


**Figure 16. Cantilever beam with rectangular cross-section**

When the beam length,  $l$ , and the Young's modulus,  $E$ , of the material are considered, the overall stiffness of the material (i.e. spring constant) can be written as:

$$k = \left(\frac{16}{5}\right) \frac{EI}{l^3} \quad (2)$$

where  $I$  is the moment of inertia for a rectangular cantilever beam described by:

$$I = \frac{wt^3}{12} \quad (3)$$

and  $w$  is the beam width and  $t$  is the thickness of the beam [24]. A change in surface stress at the end of the cantilever beam produces a bending moment,  $M$ , which can

be expressed as:

$$M = Fl \tag{4}$$

Equation 5 shows how the resonance frequency increases as the spring constant increases and as the mass of the beam decreases [24]. Expressing the resonance frequency,  $f_{res}$ , (the natural frequency of vibration determined by the physical parameters of a given material) as a function of the spring constant produces the simplified equation where mass,  $m$ , is mass density times the thickness of the beam times the length of the beam times the width of the beam  $m = m^*(t \times l \times w)$ .

$$f_{res} = \sqrt{\frac{k}{m}} \tag{5}$$

The preceding equations illustrate how basic cantilever beam operation is closely related to the mechanical stress, as it relates to material properties and geometries of a device [24][9].

### **2.3.1 Bimorph Cantilever Beams.**

Bimorph cantilever beam transducers are those with thermally-induced mechanical stresses. Bimorph cantilever beam transducers exploit the differences between the Coefficients of Thermal Expansion (CTE) of two dissimilar materials [25]. CTE is a material dependent variable, denoted as  $\alpha$ , and is defined as the amount of thermal expansion in a material, per unit length, per degree of temperature change [25]. As the material undergoes a temperature change these expanded dimensions are described by  $\Delta X = \alpha x(T - T_0)$ , where the starting temperature equals  $T_0$  and the ending temperature of the material is  $T$ . In bimorph cantilever beams, two materials are deposited at different temperatures, in contact with each other, and exposed to a change in temperature. This temperature change, coupled with the different material

properties, will cause one material to expand more than the other and induce stress into the system. This stress causes the entire structure to bend or deflect [25][9]. Figure 17 is a cross-sectional view of a simplified bimorph cantilever beam, where  $l$  is the beam length and  $E_1$  and  $E_2$  are the Young's Moduli,  $w_1$  and  $w_2$  are the widths, and  $t_1$  and  $t_2$  are the thicknesses of the specific material layers.

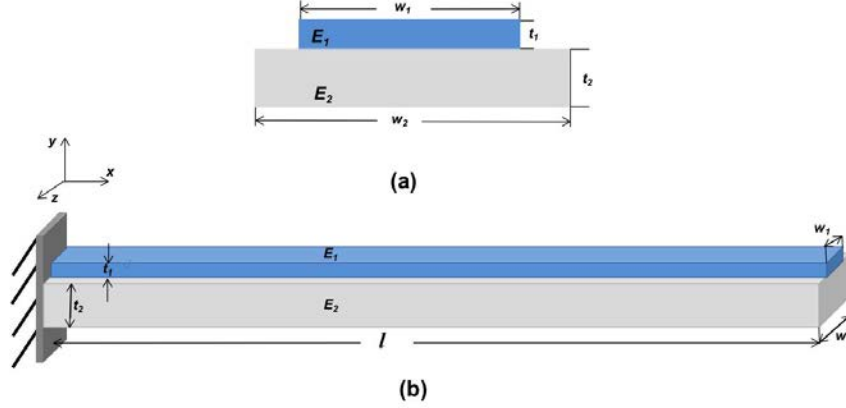


**Figure 17. Cross-sectional views of a bimorph cantilever beam: (a) front view of beam; (b) side view of beam**

[9]

A majority of bimorph cantilever beam analysis equations derived from work with bimetal thermostats and provide an expression for the radius of curvature in a bimorph cantilever beams. Equation 6 assumes the length of the two material layers to be equal and expresses the tip deflection,  $\Delta d$  (vertical displacement), as a function of the different material properties [25][9].

$$\Delta d = \frac{3l^2}{t_1 + t_2} \left[ \frac{\left(1 + \frac{t_1}{t_2}\right)^2}{3 \left(1 + \frac{t_1}{t_2}\right)^2 + \left(1 + \frac{t_1 E_1}{t_2 E_2}\right) + \left(\frac{t_1^2}{t_2^2} + \frac{t_2 E_2}{t_1 E_1}\right)} \right] (\alpha_1 - \alpha_2) \Delta T \quad (6)$$

## 2.4 Microfabricated Buckled Membranes

The goal of this section is to provide a basic understanding in the relevant theory of microfabricated buckled membranes. This will include an overview of the basics of residual stress, buckling, springs, and compliant bistable mechanisms.

### 2.4.1 Residual Stress.

In thin films, stress develops between layers primarily due to mismatched thermal expansion coefficients and growth procedure [13][6]. When a thin film is deposited on a thick substrate at elevated temperature and subsequently cooled and operated at an ambient temperature, the difference between the coefficients of thermal expansion (CTE) ( $\alpha$ ) of the silicon ( $\frac{2.5 \times 10^{-6}}{K}$ ) and silicon dioxide ( $\frac{0.55 \times 10^{-6}}{K}$ ) induces residual stress [6][13].

### 2.4.2 Buckling.

Buckling in some cases can be considered a failure mechanism causing a lateral failure of a structure as a result of the compressive force being greater than the material is capable of withstanding [13]. A common scenario of buckling is a column under a compressive load as seen in Figure 18. As the axial load on the column is increased, it reaches a critical buckling point and will bow in the direction corresponding to the lowest point of inertia [13][6].

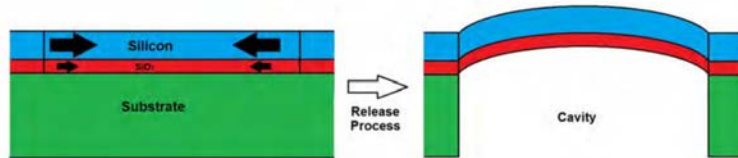


**Figure 18.** Illustration of stress-induced buckling (upon release) of the Si/SiO<sub>2</sub> layers as a result of their mismatched thermal expansion coefficients during the growth procedure [13]

## 2.5 Electroactive Polymer (EAP)

Electroactive polymers possess semi-crystalline structures in which the centers of positive and negative charges do not overlap, yielding dipoles. When subjected to mechanical vibrations, mechanical strain is applied to these materials and leads to distortion of the dipoles, creating electrical charge. The electrical energy can be harvested by storing it in capacitors or rechargeable batteries [7].

### 2.5.1 Materials.

**Table 1. Properties for selected piezoelectric ceramics, single crystals, PZT-Polymer composites, and polymers. [2]**

|                                          | PZT-5H<br>(ceramic) | PMN-32 with [001]<br>orientation (single-crystal) | PZT rod-Polymer composite<br>with 30 vol. PZT | PVDF<br>(polymer) |
|------------------------------------------|---------------------|---------------------------------------------------|-----------------------------------------------|-------------------|
| Density ( $g/cm^3$ )                     | 7.65                | 8.10                                              | 3.08                                          | 1.78              |
| Dielectric constant $\epsilon_r$         | 3250                | 7000                                              | 380                                           | 6.0               |
| Young's modulus $Y_{33}$ (GPa)           | 71.4                | 20.3                                              | -                                             | 2                 |
| Mechanical quality factor $Q_m$          | 32                  | -                                                 | -                                             | 10                |
| Piezoelectric charge constant d33 (pC/N) | 590                 | 1620                                              | 375                                           | 25                |
| Piezoelectric charge constant d31 (pC/N) | -270                | -760                                              | -                                             | 12-23             |
| Electro-mechanical coupling factor k33   | 0.75                | 0.93                                              | -                                             | 0.22              |

The following electroactive polymers have demonstrated piezoelectric, pyroelectric or ferroelectric properties: Nylon-11 [26], polylactic acid (PLLA) [27], poly(lactic-co-glycolic acid) (PLGA) [28], and poly(vinylidene fluoride) (PVDF) [7]. Table 1 shows the properties of several piezoelectric-based compounds. However, PVDF and its copolymers have demonstrated the best all-around electroactive properties [28][7]. Many of the interesting properties of PVDF, in particular those related to its use as a sensor or actuator, are related to the strong electrical dipole moment of the PVDF which results from the electronegativity of fluorine atoms as compared to those of hydrogen and carbon atoms [28][5]. In this way, each chain possesses a dipole moment perpendicular to the polymer chain. This semi-crystalline polymer shows a complex structure and can present several distinct crystalline phases related to different chain conformations. As shown in Figure 19, the  $\beta$ -phase possesses the

highest dipole moment per unit cell when compared to the other two phases ( $\alpha$  &  $\gamma$ ) and is therefore the most responsive piezoelectric polymer.

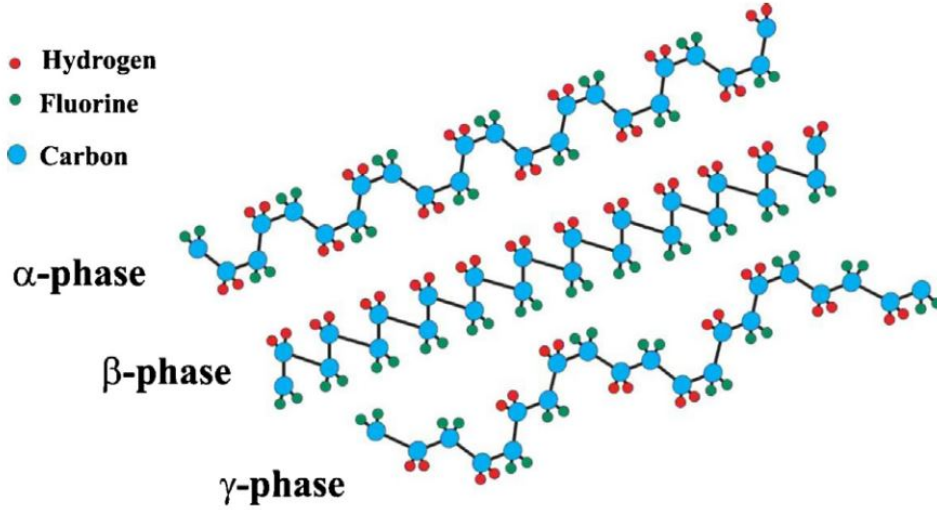


**Figure 19.** Schematic representation of the chain conformation for the  $\alpha$ ,  $\beta$ , and  $\gamma$  phases of PVDF

[28]

Previous processes used to manufacture PVDFs piezoelectric  $\beta$ -phase have been limited to drawn films. Therefore in order to obtain the electroactive phases of PVDF, different strategies have focused on the inclusion of specific copolymers such as P(VDF-TrFE) [5][29][15][28]. As shown in Fig. 20, P(VDF-TrFE) always exhibits the ferroelectric  $\beta$  crystalline phase [7][28]. The fluorine atom from TrFE stabilizes the  $\beta$ -crystalline phase and discourages  $\alpha$ -crystalline phase formation [7]. This property permits P(VDF-TrFE) copolymer to be produced in the form of thin-films by spin coating, and allows a suitable control of sample thickness which is ideal for the production of energy-harvesting microstructures.

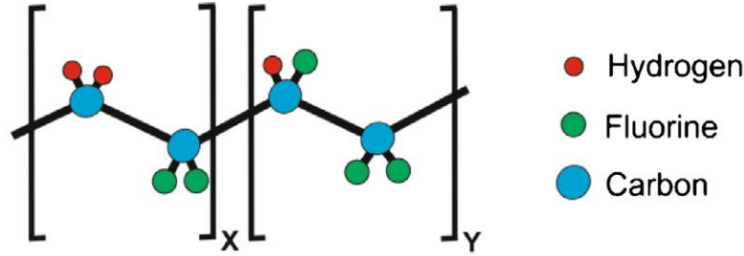


Figure 20. Schematic representation of the P(VDF-TrFE) repeat units

[28]

### 2.5.2 Polarization.

As briefly discussed above, the ferroelectric crystalline phase or  $\beta$  phase of P(VDF-TrFE) is responsible for the electroactive phase in the material presented in this research. Therefore the  $\beta$  phase can be spontaneously polarized in which polarization can be reversed by applying an electric field. An important characteristic of ferroelectric material is called a domain or dipole moment in P(VDF-TrFE). A domain is a microscopic region of a crystal in which the polarization is homogeneous. These domains (or dipoles) are naturally un-aligned, but can be aligned by heating the electroactive polymer at its curie point and applying a DC electric field for an extended period of time ( $\sim 105^\circ\text{C}$  and  $26\text{V}/\mu\text{m}$  for P(VDF-TrFE) thin films [1]).

## 2.6 Finite Element Modeling

FEM in conjunction with Computer-Aided Design (CAD) software is used by designers to model and simulate complex structures. FEM cuts a structure into several smaller elements (pieces of the structure) then reconnects elements at nodes as if nodes were pins or drops of glue that hold the elements together. It accomplishes this difficult task by first characterizing smaller elements of the structure then combines those results into a single model and/or simulation set of simultaneous algebraic

equations. MEMS design and fabrication often leverages FEM tools to increase the competence of Research and Development (R&D). These tools allow the modeling and simulation of new MEMS designs prior to fabrication increasing confidence and reducing multiple costly fabrication runs. There are many FEM tools available, but in MEMS design and fabrication there are three which appear most often ANSYS<sup>®</sup>, COMSOL Multiphysics<sup>®</sup>, and CoventorWare<sup>®</sup>.

ANSYS<sup>®</sup> is a comprehensive software suite expanding the entire range of physics, which simulates virtually all engineering fields. This tool provided designs scalability, from macro to micro, of the comprehensive multiphysics foundation and architecture. The ANSYS suite enables simulation of a wide range of sensors and actuators, from RF sensors dependent on electromagnetic fields to gyroscopes dependent on mechanical motion, as well as piezoelectric devices with both mechanical and electromagnetic components. ANSYS<sup>®</sup> Inc recently partnered with Tanner Electronic Design Automation (EDA) to include the ability to import 3D MEMS Pro files from Tanner EDAs L-Edit design layout editor, a common tool used by MEMS designers which is discussed in the following chapter. ANSYS's workbench framework also provides the user a simple drag-and-drop interface, with bi-directional parametric CAD software, automated meshing and project-level updates, pervasive parameter management, and integrated optimization tools [30][31][9].

COMSOL Multiphysics<sup>®</sup> is a multiphysics FEM is a general-purpose software platform for electrical, mechanical, fluid flow and chemical applications. The COMSOL Multiphysics<sup>®</sup> multipurpose software platform is ideally suited for MEMS applications and contains the best-in-class piezoelectric and piezoresistive FEM tools currently available. It includes modules for analyzing stationary and transient domains, fully-coupled eigenfrequency, parametric, quasi-static, and frequency responses. COM-

SOL Multiphysics also allows the user to perform lumped parameter extraction of capacitance, impedance, and admittance on MEMS designs [32][33][9].

CoventorWare<sup>®</sup> is a FEM tool used to predict complex, multiphysics behaviors in MEMS devices, with a focus in MEMS-specific capabilities. It is ideal for simulating the mechanical, electrostatic, piezoelectric, piezoresistive and thermal effects for MEMS sensors and actuators. The CoventorWare<sup>®</sup> suite is capable of simulating devices' sensitivity, linearity, frequency response, signal-to-noise ratio, temperature stability, and actuation times. Coventor<sup>®</sup> has also partnered with MEMSCAP<sup>®</sup> Inc and Tanner EDA to include MUMPs<sup>™</sup> specific material properties in its material library as well as the ability to import design files from Tanner EDAs L-Edit design layout editor. Additionally, CoventorWare<sup>®</sup> provides designers a 2D-to-3D model builder, automatic meshing, and field solvers [34][9].

Each of these FEM tools provides a multiphysics-based platform with the capability of simulating MEMS devices. Although slightly different, they are all capable of performing the unique multiphysics challenges which come with the simulation of MEMS devices.

## 2.7 Summary

This chapter presented a background literature review of previous and current research pertaining to MEMS micro-mirror design, fabrication, and applications. Various microfabrication procedures and techniques were introduced, covering the three types of fabrication techniques common to MEMS fabrication, and commercially available fabrication options. These topics were followed by a brief review of transducers, highlighting the differences and providing simple examples for sensors and actuators. The final section of this chapter provided an extensive review of current micro-mirror research, covering electrostatically-, electrothermally-, and

piezoelectrically-actuated micro-mirror assemblies. The information presented in this chapter builds a foundation of understanding for research topics covered in the remainder of this thesis.

## III. METHODOLOGY

### 3.1 Chapter Overview

The previous chapter outlined the basic theory behind the behavior of electroactive polymer films studied in this research. This chapter presents a novel design approach, analytical modeling, finite element modeling, Membrane device fabrication, PolyMUMPs post-fabrication processing, tools and equipment, and device characterization methodologies used in the evaluations of this unique design solution.

#### 3.1.1 P(VDF-TrFE) Preparation for Processing.

As previously discussed in Chapter 2.2, spin coating is commonly used in micro-fabrication, which allows depositing a thin layer of material on a substrate typically with a thickness in the range of a few nanometers to a few hundreds of micrometers. Most commonly, a drop of the resist dissolved in a solvent is deposited on a substrate which can be then spread to cover the entire surface of the substrate by spinning [4]. During the spinning, the solvent will be partially evaporated, resulting in a thin film coated on the substrate. The remaining solvent will be further evaporated by subsequent heating or annealing steps.

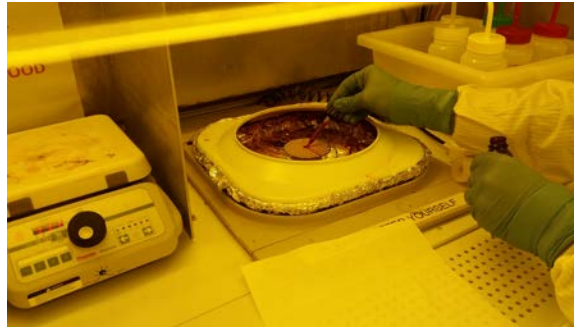
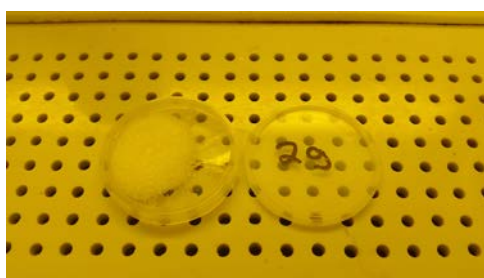


**Figure 21.** Illustration of spin coating used throughout processing of devices presented in this research

Typically P(VDF-TrFE) is dissolved with a 5-15% concentration in polar solvents, such as listed in Table 2. In this research 8%-15% PVDF dissolved in methyl ethyl ketone (MEK) was used mainly due to the availability of the solvent. However, P(VDF-TrFE) is often dissolved in a mixture of 2 solvents: one with a slow rate of evaporation (DMF, DMAc or DMSO) which solutes the polymer, and another one with a fast rate of evaporation (i.e. acetone), as listed in Table 3, which typically evaporates during the spin-coating [4][35][28][36][37]. Figure 22 shows the processing of P(VDF-TrFE) powder for spin coat solution.

**Table 2. Table of P(VDF-TrFE) Active Solvents [3][4]**

| Solvent                   | Boiling Point (°C) | Flash Point (°C) | Polarity Index |
|---------------------------|--------------------|------------------|----------------|
| Tetrahydrofuran           | 65                 | -17              | 4.0            |
| Methyl Ethyl Ketone       | 80                 | -6               | 4.7            |
| Tetramethyl Urea          | 177                | 65               | 5.0            |
| Dimethyl Formamide (DMF)  | 153                | 67               | 6.4            |
| Dimethyl Acetamide (DMAc) | 166                | 70               | 6.5            |
| N-Methyl-2-Pyrrolidone    | 202                | 95               | 6.7            |
| Dimethyl Sulfoxide (DMSO) | 189                | 35               | 7.2            |
| Triethyl Phosphate        | 195                | 107              | -              |



(a) 2g P(VDF-TrFE) powder



(b) 25ml of P(VDF-TrFE) solution

**Figure 22. Preparation of 8% weight/volume MEK:P(PVF-TrFE) solution. Combined, covered with foil to lessen evaporation, then placed on magnetic stir hotplate (150RPM) for 3hrs at 70°C followed by 8hrs stir at ambient**

**Table 3. Table of P(VDF-TrFE) Latent Solvents [3]**

| Solvent                | Boiling Point (°C) | Flash Point (°C) |
|------------------------|--------------------|------------------|
| Acetone                | 56                 | -18              |
| Methyl Isobutyl Ketone | 118                | 23               |
| Glycol Ethers          | 118                | 40               |
| Glycol Ether Esters    | 120                | 30               |
| N-Butyl Acetate        | 135                | 24               |
| Cyclohexanone          | 157                | 54               |
| Diacetone Alcohol      | 167                | 61               |
| Diisobutyl Ketone      | 169                | 49               |
| Ethyl Aceto Acetate    | 180                | 84               |
| Butyrolactone          | 204                | 98               |
| Isophorone             | 215                | 96               |
| Triethyl phosphate     | 215                | 116              |
| Carbitol Acetate       | 217                | 110              |
| Propylene Carbonate    | 242                | 132              |
| Glyceryl triacetate    | 258                | 146              |
| Dimethyl Phtalate      | 258                | 149              |

### 3.2 Design

Several applications have been identified where free, unused mechanical energy could be used to generate electrical energy and are summarized in Table 4. Various

**Table 4. Frequency and acceleration of various vibration sources [5]**

| Vibration source              | Frequency (Hz) | Acceleration amplitude ( $m/s^2$ ) |
|-------------------------------|----------------|------------------------------------|
| Car instrument panel          | 13             | 3                                  |
| Casing of kitchen blender     | 121            | 6.4                                |
| Clothes dryer                 | 121            | 3.5                                |
| HVAC vents in office building | 60             | 0.2-1.5                            |
| Car engine compartment        | 200            | 12                                 |
| Refrigerator                  | 240            | 0.1                                |
| Human walking                 | 2-3            | 2-3                                |

solutions exist to improve energy harvesting efficiency. For instance we could increase the area of the P(VDF-TrFE) copolymer, polarization of the copolymer, as well as the strain and/or frequency imposed on the energy harvesting structure. To overcome

the complexity of combining all these design capability requirements into a single device, each requirement was considered individually. This approach allows each design choice to be chosen to maximize the performance of the capability itself while considering the effect it has on other possible choices.

The first design decision made is to pursue the fabrication and testing of a basic metal-insulator-metal device, as illustrated in Figure 23(a), for purposes of characterizing the steps involved in processing P(VDF-TrFE). The device shown in Figure 23(b) will be used to characterize the electroactive properties of the P(VDF-TrFE). This decision allows the research to initially focus on the processing and fabrication first since device performance relies on the ability to effectively process and pole the P(VDF-TrFE) for energy harvesting application.

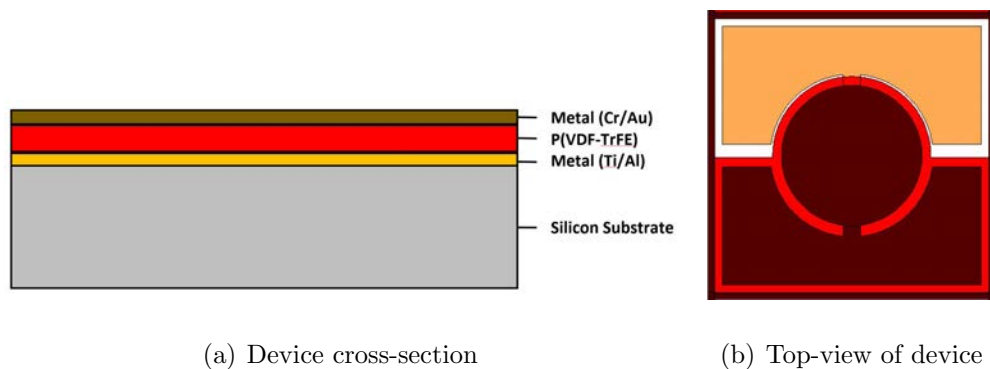


**Figure 23. Basic electro-active polymer (EAP) metal-insulator-metal device for P(VDF-TrFE) characterization**

In conjunction with the basic EAP device shown in Figure 23, the processing of both membrane and PolyMUMPs devices are also explored. Figure 24 illustrates the membrane device fabricated in this research. It is assumed that a larger deflecting surface will yield greater energy production when applied for energy harvesting. From prior research it is shown that when comparing the differently-sized membranes of the same thickness, it was found that as the size of the membrane increases, so does its deflection over a given temperature range [6]. Therefore a membrane with a

lower critical stress will deflect more than a membrane with a higher critical stress for the same applied temperature. The relation between the membrane size and its deflection for the same applied temperatures is summarized in Table 5. Ultimately these principles have been taken into consideration during the design phase.

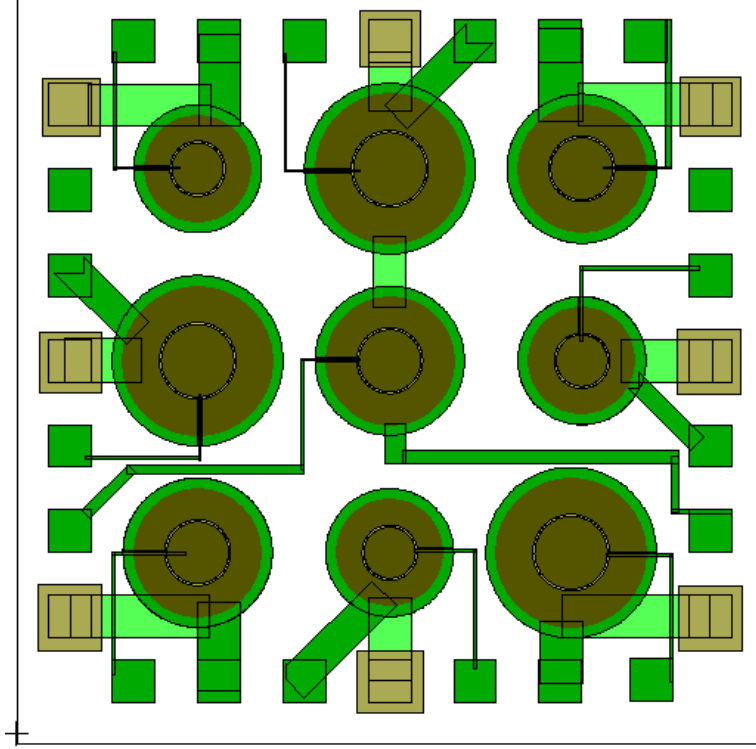


**Figure 24.** Membrane (metal-insulator-metal) energy-harvesting device structure with non-segmented bottom electrodes and segmented (inner/outer) top electrodes to capitalize harvesting for both tensile and compressive failure modes of membrane buckling

**Table 5.** Table of membrane deflection results for FEM modeling and measured data for a membrane thickness of  $5\mu\text{m}$  at the same applied temperatures [6]

| Membrane Diameter ( $\mu\text{m}$ )   | 1,000 | 1,500 | 2,000 |
|---------------------------------------|-------|-------|-------|
| Measured Deflection ( $\mu\text{m}$ ) | 9.30  | 17.50 | 24.80 |
| FEM Deflection ( $\mu\text{m}$ )      | 10.46 | 19.60 | 27.78 |

The PolyMUMPs<sup>®</sup> fabrication process will be used to fabricate the MEMS sub-structure shown in Figure 25 and will provide functional MEMs prototypes for this research. This allows not only allows study on the functional design, but allows

for focus on the fabrication and required post-processing of such Multi-User MEMS Process (MUMPs) devices. The MUMPs commercial process affords time for three fabrication opportunities during the research time-line. It also solidified specific initial design constraints which provided a starting point for the design.

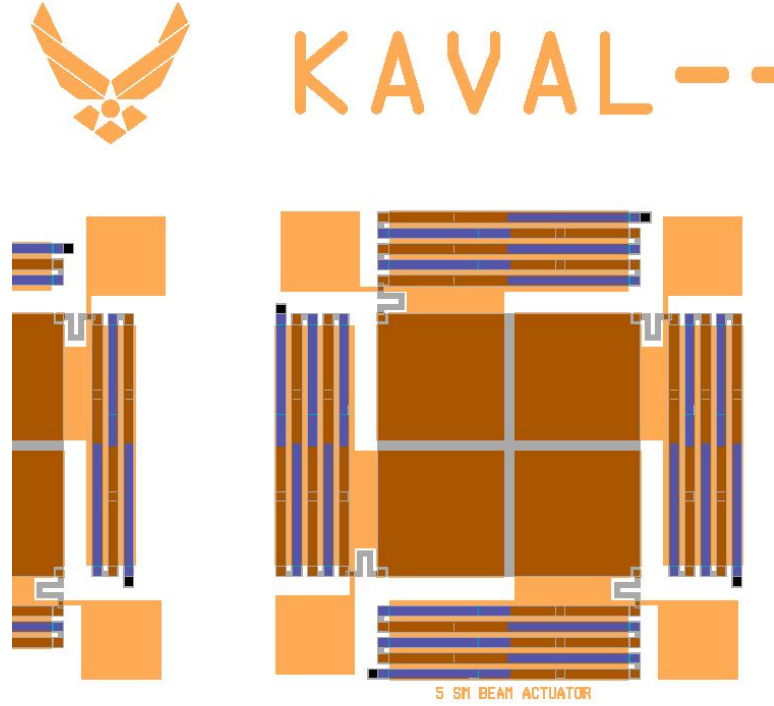


**Figure 25. PolyMUMPs micro-mirror platform which will require post-processing of metals & P(VDF-TrFE) (deposition and patterning) prior to HF release**

Since EAP are also capable of converting energy in the form of electric charge and voltage to mechanical force and movement, there is potential to utilize these same devices as transducers (actuator and sensor). This principal could be applied to provide alternate actuation and sensing schemes as discussed in Chapter 6.

### **3.2.1 Cantilever Initial Deflections.**

In an at-rest state, the partial-bimorph transducer is deflected upward (deformed). The methodology for calculating the maximum initial deflection of a partial-bimorph

cantilever beam is presented here; to do this the beam will be analyzed in two parts. Part one uses Equation 6 and provides the portion of the beam deflection resulting from the bimorph segment of the cantilever beam,  $d_1$ . Part two calculates the deflection of the non-bimorph segment of the beam,  $d_2 = l_2 \sin \theta$  [9]. Figure 26 is a cross-sectional view of a partial-bimorph cantilever beam illustrating how  $d_2$  is to be calculated. Therefore the total deflection can then be calculated as  $\Delta d = d_1 + d_2$ .

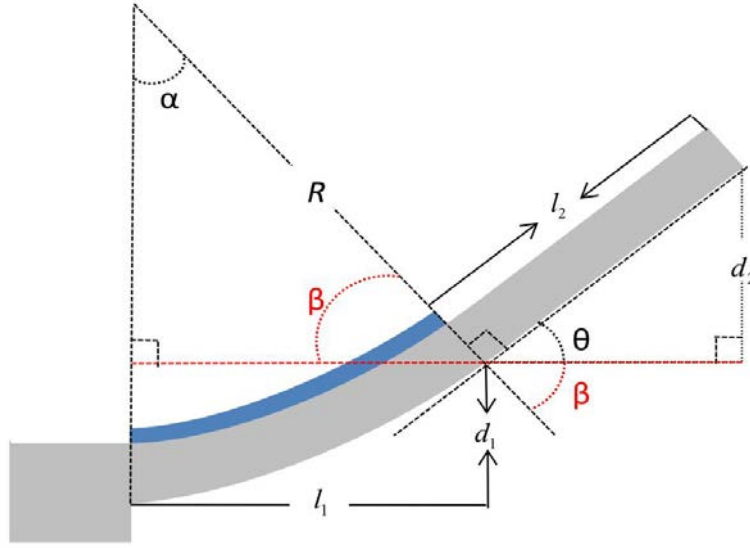


**Figure 26. Cross-sectional view of a partial bimorph cantilever beam illustrating how initial beam deflection will be calculated**

[9]

A model representing a bimorph energy harvesting structure was designed in simulation taking into account the additional deposition steps involved. Finite element modeling was conducted using CoventorWare<sup>®</sup> to evaluate the addition of PVDF and top gold electrode deposition (post PolyMUMPS processing). More specifically, partial bimorph cantilever beam material properties and geometries were evaluated. In modeling and simulation, the addition of PVDF and gold reduced the deflection by approximately 66% independent of actuator length.

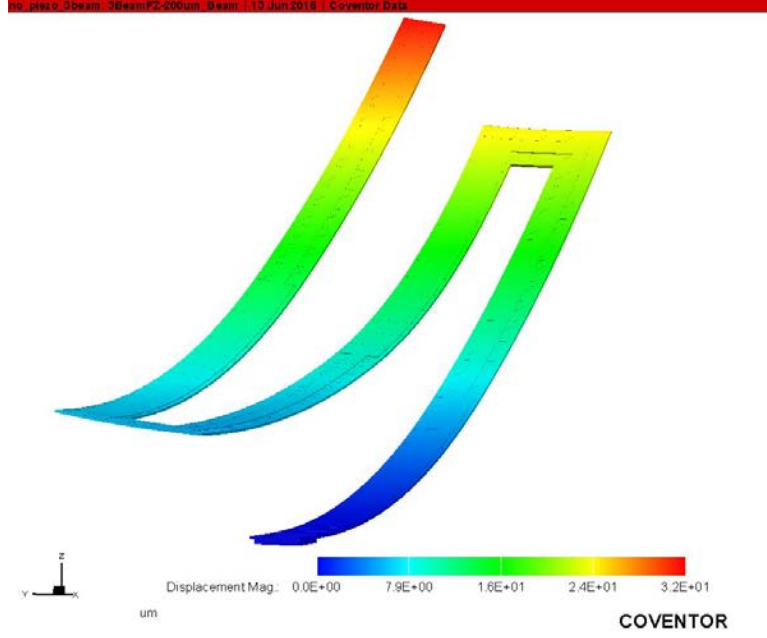


**Figure 27.** Example of simulated post-release ('at-rest' state) of partial-bimorph structure with additional post-processing steps to include deposition & patterning of P(VDF-TrFE) and metal electrode layers

### 3.2.2 Resonant Frequency.

Harvester performance can be optimized to specific applications provided a known resonant frequency from Figure 28, which is given by:  $\omega_{nf} = \alpha_n^2 \sqrt{\frac{EI}{mL^4}}$  where  $\alpha_n = 1.875, 4.694, 7.885$  [38]. Therefore the resonance frequency of a simply supported cantilever beam can be calculated using Equation 7-8 where  $E$  is the Young's modulus,  $I$  is the moment of inertia,  $L$  is the length of the cantilever,  $w$  is the width of the cantilever,  $m$  is the mass per unit length of the cantilever beam, and  $\alpha_n = 1.875$  is the eigenvalue for the fundamental vibration mode [7].

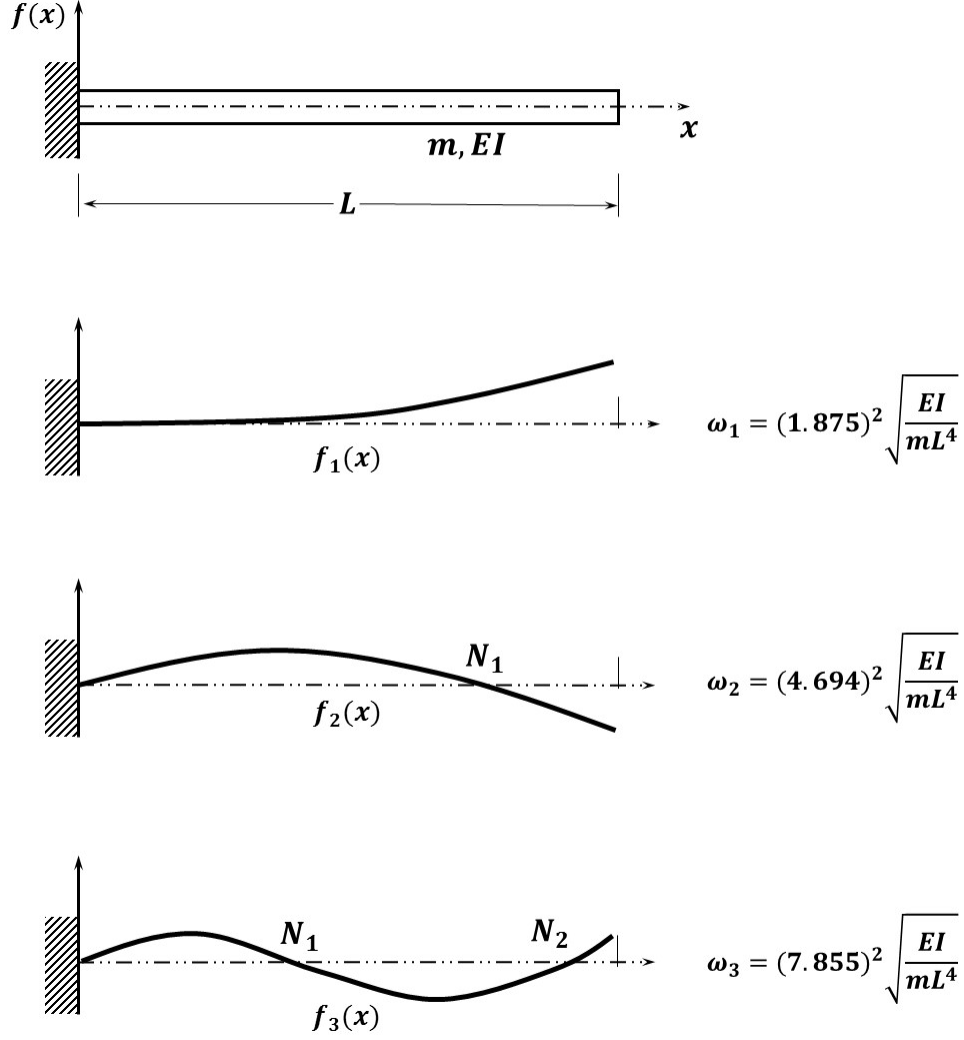


Figure 28. The first three undamped natural frequencies and mode shapes of a cantilever beam

$$f_r = \frac{\alpha_n^2}{2\pi} \frac{1}{L^2} \sqrt{\frac{EI}{mw}} \quad (7)$$

$$f_r = \frac{\alpha_n'^2}{2\pi} \frac{1}{L^2} \sqrt{\frac{K}{m_e + \Delta m}} \quad (8)$$

In order to further lower the resonance frequency of the cantilever microstructure, a proof mass can be attached to the free end of the cantilever (Figure 29). In which

case (7) can be approximated by (8), to include the proof mass  $\Delta m$  where  $\alpha_n'^2 = \alpha_n^2 \sqrt{0.236/3}$ ,  $m_e = 0.236mwL$  is the effective mass of the cantilever, and  $K$  is the effective spring constant of the cantilever [7][39].

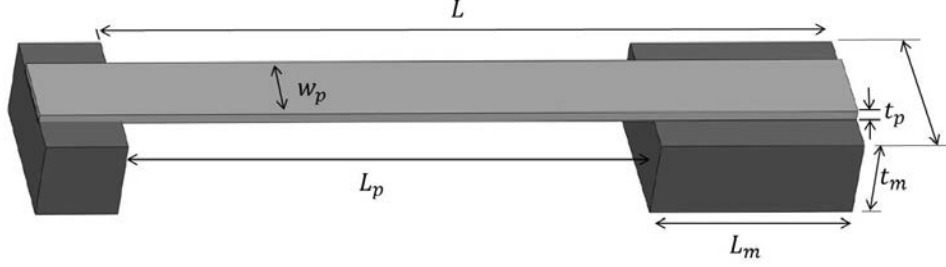


**Figure 29. Proof mass attached to the free end of a cantilever beam**

### 3.3 Device Fabrication

As previously stated, fabrication of these device designs was accomplished both commercially and in-house. The commercial device fabrication was conducted using PolyMUMPs. The MUMPs fabrication processes afforded three design fabrication opportunities during the time allotted for this research. Each fabrication run consisted of 15 copies of one  $1cm \times 1cm$  area of design space called a die site. MEMSCAP® required that each die site layout be designed using Tanner EDAs L-Edit MEMS software. L-Edit MEMS layout editor software was created by Tanner EDA and designed specifically to simplify the difficult task of preparing a design layout for fabrication.

#### 3.3.1 Membrane Fabrication and Processing.

The buckled membrane structures are fabricated on a SOI wafer. The wafers consist of a  $500\mu m$  thick Si handle with a  $2\mu m$  thick buried oxide layer of  $SiO_2$  grown on top of the handle layer. A  $5\mu m$  thick device layer of Si is bonded on top of the buried oxide layer. The membranes are released by way of a backside etch through

the entire depth of the handle wafer, stopping at the buried SiO<sub>2</sub> as seen in Figure 30&31 [13].

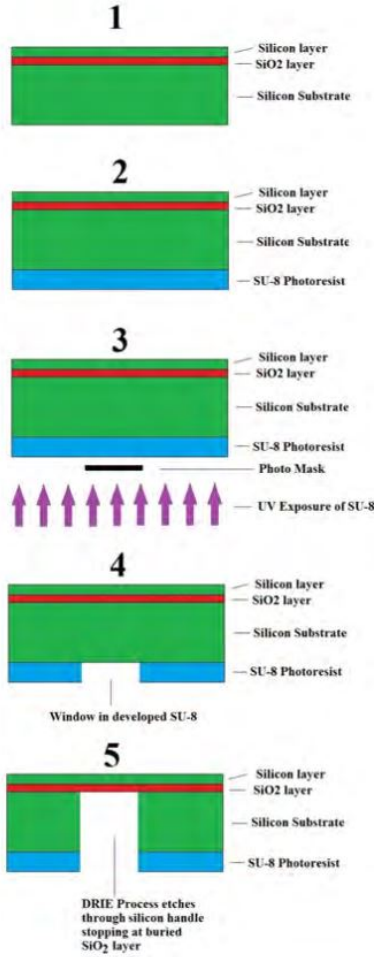


**Figure 30.** Illustration showing the fabrication process for the buckled membrane [13]

Prior to the DRIE step the SOI wafers were processed as illustrated in Figure 32. First the samples were submerged in a buffered oxide etch (BOE) for 1 minute to remove any native oxide and maximize SU-8 adhesion in subsequent processing steps. The samples were then rinsed in DIW for 30 seconds and dried with pressured nitrogen gas (N<sub>2</sub>). The samples were cleaned with a 30-second acetone rinse, 30-second methanol rinse, and a 30-second deionized water (DIW) rinse and dried with pressurized N<sub>2</sub>.

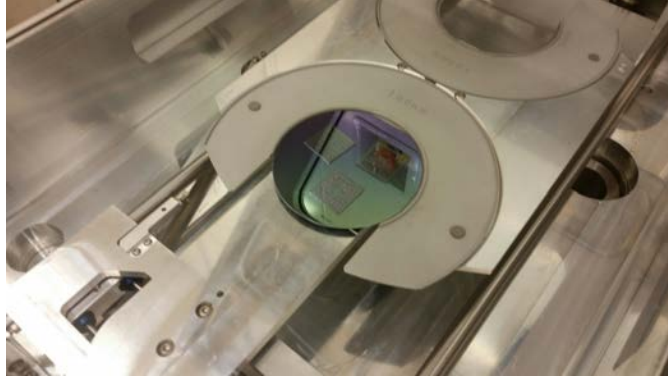


**Figure 31. DRIE fabrication process step for the buckled membrane using University of Michigan's STS Pegasus DRIE tool**

A layer of S1818 photoresist was spin coated onto the device layer side of the sample and spun for 30 seconds at 4000 RPM resulting in a  $1.8\mu m$  thick coating of photoresist. This was then baked at  $110^{\circ}C$  hotplate for 75 seconds and then allowed to cool. The photoresist is then exposed to UV light using the EVG mask aligner for 7 seconds to provide an exposure dose of  $85mJ/cm^2$ . Following the exposure, the photoresist was developed in a developer solution of 5:1 DI water to 351 developer for 45 seconds. Following the development, the sample was rinsed in DI water for 30 seconds and dried with pressurized  $N_2$ .

With the bottom electrodes now patterned in the photoresist, the samples were placed in the Torr International electron beam evaporation tool. This tool uses a focused beam of electrons to evaporate metals which then deposit on the samples. The first layer deposited was a  $50\text{\AA}$  layer of titanium (Ti) to serve as an adhesion layer between the silicon and the  $3000\text{\AA}$  aluminum (Al) which was subsequently deposited.

After the the metal deposition, unwanted metal was removed by a liftoff process. The samples were placed in a container of acetone which was then placed into an ultrasonic bath. This acetone dissolved the remaining photoresist which removed the metal deposited on top of it, leaving only the bottom electrodes on the sample.

Next, an 8% (weight/volume) P(VDF-TrFE) copolymer solution was deposited by spin coating at 2000 rpm for 30 seconds which resulted in a  $1\mu m$  thick coating of copolymer. The sample was then baked at  $100^{\circ}C$  hotplate for 5 hours. The hotplate was turned off with the sample remaining and allowed to cool slowly until the sample was within  $\pm 5^{\circ}C$  of ambient.

Another patterned photoresist layer was used as an RIE mask to pattern the P(VDF-TrFE) thin film. A layer of S1818 photoresist was spin coated on to the device layer side of the sample and spun for 30 seconds at 4000 RPM resulting in a  $1.8\mu m$  thick coating of photoresist. The photoresist was then exposed and developed in a developer solution of 5:1 DI water to 351 developer for 30 seconds. Following the development, the sample was rinsed in DI water and dried with pressurized  $N_2$ . The RIE dry etch was performed under 100 Standard Cubic Centimeters per Minute (SCCM) oxygen gas environment with 200W Radio Frequency (RF) power and  $5mT$  pressure. The P(VDF-TrFE) thin film etch rate was 150nm/min. The photoresist mask was etched simultaneously with an etch rate of 100nm/min and dry etch was continued until the photoresist mask was fully etched.

For the top electrode, a  $30\text{\AA}$  layer of chromium was deposited followed by a  $2000\text{\AA}$  layer of gold. A layer of S1818 photoresist was spin coated onto the device layer side of the sample and spun for 30 seconds at 4000 RPM. The photoresist was then exposed and developed. Following the development, the sample was rinsed in DI water and dried with pressurized  $N_2$ . The top Ti/Al electrode ( $2500\text{\AA}$  thick) was patterned by wet etch and the photoresist mask was again removed by RIE dry etch.

A layer of SU-8 was spin coated at 2800 RPM for 30 seconds on the handle side of the sample to obtain a  $\sim 17\mu m$  thick coating of SU-8. Following the spin coating, the sample was soft baked on a  $65^{\circ}C$  hotplate for 2 minutes then placed on a  $95^{\circ}C$  hotplate for 5 minutes. The samples were then allowed to cool before they were

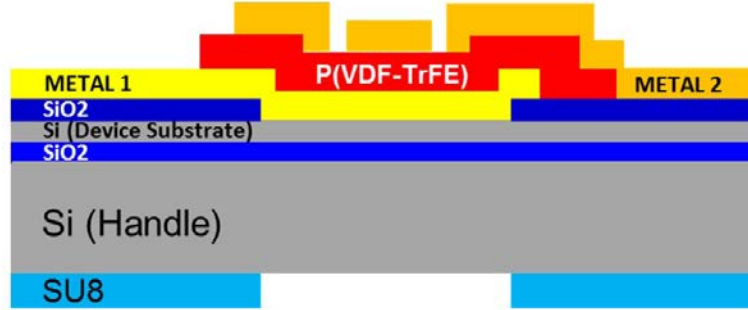
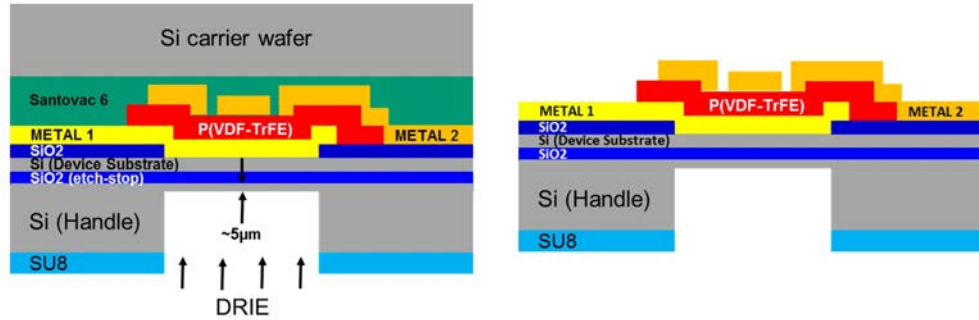


**Figure 32.** Additional layers and respective thickness of processing steps for the buckled membrane device

exposed. The samples were aligned using the EVG mask aligner and exposed. The UV power of this tool is set to  $11mW/cm^2$ . The exposure time was set to 15 seconds to provide an exposure energy of  $165mJ/cm^2$ . Following the exposure, the samples were placed on a  $65^\circ C$  hotplate for 1 minute then placed on a  $95^\circ C$  hotplate for 5 minutes for the post exposure bake (PEB). The samples were then developed for 1 minute using Microchem's SU-8 developer and then rinsed in DIW. The development opens up the windows in the SU-8 mask layer that will allow the cavity in the handle to be etched with DRIE. Santovac 5 polyphenyl ether was used to attach the sample to a carrier wafer for the DRIE process as seen in Figure 33. Sample mounting or temporary bonding was used to laminate the device substrate side to a handle wafer. This step was necessary because the sample was smaller than the tool chuck (3" wafer on a 6" chuck) in addition to mechanical support being required for the material during processing. The SPTS Pegasus 6 deep reactive ion etch (DRIE) tool was used to etch the SOI device wafers using the Bosch process. The Pegasus 6 DRIE tool has a high power ICP source capable of fast (over  $20\mu m/min$ ), high aspect ratio

(up to 100:1) anisotropic etching of silicon. Upon completion of DRIE processing to within  $\pm 5\mu\text{m}$  of the  $\text{SiO}_2$  etch-stop, the sample was separated from the carrier wafer by soaking in methanol for 12hrs followed by a DIW rinse as illustrated in Figure 34.



(a) SOI device Pegasus 6 DRIE processing (b) SOI device post DRIE processing

**Figure 33. (a) SOI device DRIE processing:** sample attached to a carrier wafer for mechanical support using Santovac 5 polyphenyl ether prior to DRIE processing; **(b) Post DRIE processing:** sample was separated from the carrier wafer after a 12hr soak in methanol

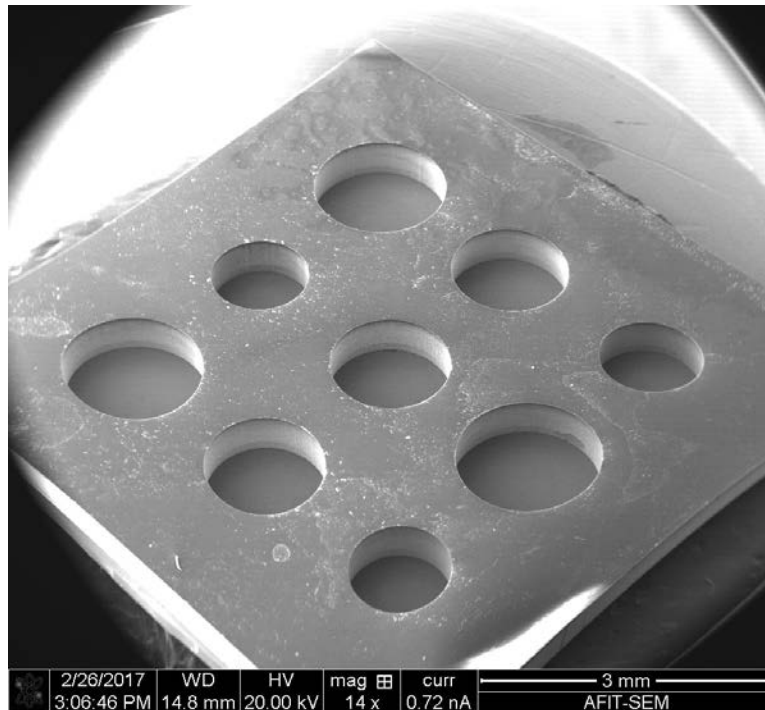


**Figure 34. SEM image of SOI device sample following DRIE, 12hr methanol soak, and separation from the carrier wafer**

### 3.3.2 PolyMUMPs Post Fabrication Processing.

Understanding and accepting the limitations of the PolyMUMPs fabrication processing, post-process fabrication of these devices were conducted in the Air Force Institute of Technology (AFIT) class 100 clean-room located on Wright-Patterson Air Force Base, Ohio. Post-fabrication processing activities followed the same process as described in the previous section with the exception of the SU-8 and DRIE processing steps. Figure 35 shows the processing steps involved in the deposition and patterning of the PolyMUMPs devices.

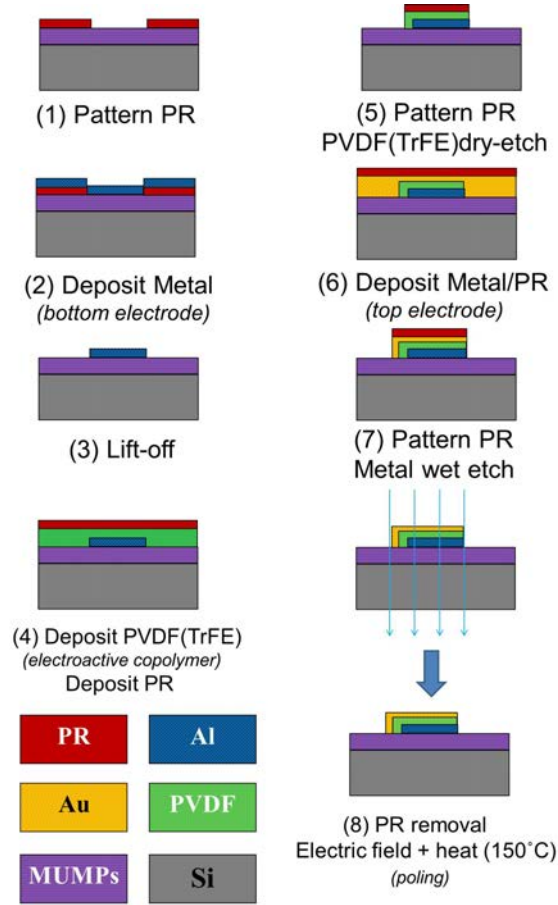


Figure 35. PolyMUMPs post-processing fabrication steps involving the deposition and patterning of PVDF films

### 3.4 Tools and Equipment

Other than device fabrication conducted through PolyMUMPs and at AFRL facilities, all device fabrication, characterization, and testing were conducted in AFITs Cleanroom and Device Characterization Lab. For this there were four main pieces of equipment used: Quanta 3D 200i Scanning Electron Microscope, used to image devices; a Micromanipulator probe station, used to optically observe device condition and sensing; EVG<sup>®</sup>620 Automated Mask Alignment System and Karl Suss MJB3 Mask Aligner, used during the post-processing photolithography steps; and a 3D optical surface profiler, ZYGO<sup>®</sup> NewView<sup>™</sup> 7300 white light interferometer, to measure initial device deflections.

The first two pieces of equipment were used throughout this research to provide visual verification of the device condition and provide the high quality imagery of the fabricated devices. The EVG<sup>®</sup>620 Automated Mask Alignment System was used during the initial fabrication stages of this research while the MJB3 mask aligner was exclusively used during the PolyMUMPs post-fabrication portion of this research. These tools were used to align the pattern transfer masks to expose the photoresist layer to ultraviolet (UV) light. The final piece of equipment used was by far the most important piece of equipment to this research. The Micromanipulator probe station is a analytical probe station designed for low current, sub-micron positioning applications, with a thermal chuck, unique probe holders, vibration isolation and camera systems used for characterizing ferroelectric/electro-active polymer films.

### 3.5 Device Characterization and Testing

Fabricated devices were evaluated in two parts, using the ZYGO NewView 7300 series white light interferometer. First the initial deflections for devices were characterized, recording initial deflection height measurements for all membrane devices

and MUMPs micro-mirror platforms. In addition to the device characterization conducted in this research, the devices were also operationally tested. The purpose for this testing was to characterize the energy harvesting performance for each device.

### 3.5.1 Piezoelectric Characterization.

Ferroelectric materials are categorized as materials that have electric dipole moments, i.e. electric polarization (charge per unit area), in the absence of an external electric field. The polarization can be reversed by the application of electric field. Thin film ferroelectrics require only few volts to change the polarization of the material. Piezoelectric materials are a subset of ferroelectric materials, where there is a relation between mechanical stress and voltage. Due to the stress-electric field coupling, piezoelectric materials are used in variety of applications such as sensing and actuation on multiple length scale (nano to macro-scale).

In this experiment to characterize the materials, voltage is applied across the sample, while the output voltage is measured across a reference capacitor. The result is a Polarization-Electric Field (P-E) hysteresis loop of tested piezoelectric sample. The Sawyer-Tower circuit shown in Figure 36 is used to measure ferroelectric hysteresis by placing the sample capacitor ( $C_S$ ) in series with a reference capacitor ( $C_{ref}$ ) of a much larger capacitance.

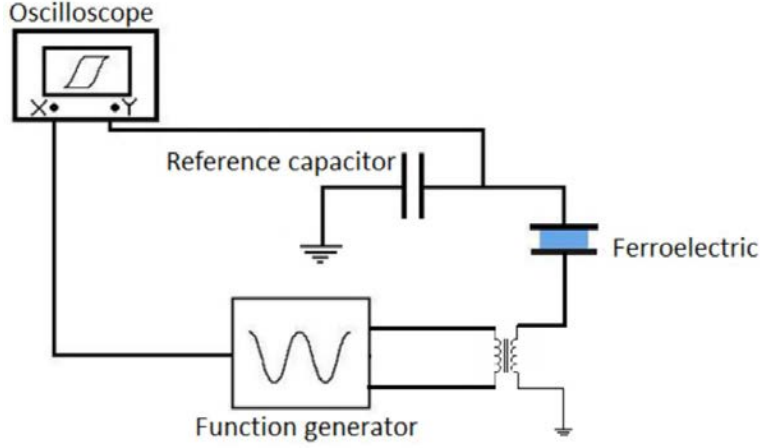


Figure 36. The Sawyer-Tower circuit

36

If the capacitance of the reference capacitor ( $C_{ref}$ ) is much larger than the capacitance of the test sample ( $C_S$ ), the voltage drop across the reference capacitor can be neglected, taking the voltage across the sample to be nearly equal to the voltage from the function generator (source). The input voltage and the thickness of the test sample are then used to calculate the applied electric field, i.e. input field where  $E$  is the electric field and  $t$  the test sample thickness.

$$E = \frac{V_{input}}{t} \quad (9)$$

To construct the P-E hysteresis loop of piezoelectric sample, the voltage across the reference capacitor ( $V_{output}$ ) in Sawyer-Tower circuit is measured and the electric polarization is calculated where  $Q$  is the charge,  $P$  is polarization, and  $A$  is the sample surface area.

$$Q = \frac{V_{output}}{C_{ref}} \quad (10)$$

$$P = \frac{Q}{A} \quad (11)$$

Figure 37 shows a typical P-E hysteresis loop, where: remanent polarization is the polarization at zero electric field, saturation polarization is indication that all material dipole moments are aligned with electric field, and coercive electric field is electric field at zero-net polarization (areas of each type of domain are equal).

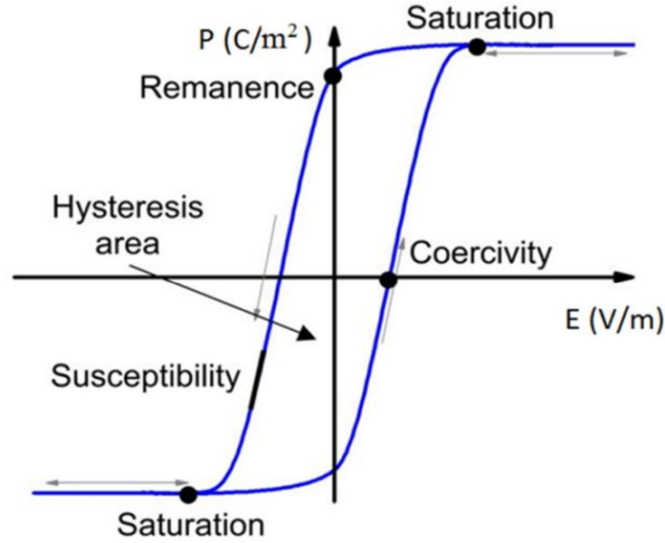


Figure 37. Typical P-E hysteresis loop

[40]

As mentioned,  $C_{ref}$  has to be much larger than  $C_s$ , in this experiment let  $C_{ref} = 1000C_s$ . The sample capacitance can be calculated

$$C_s = \frac{\varepsilon_r \varepsilon_0 \pi r^2}{t} \quad (12)$$

where,  $\varepsilon_r$  is the material relative permittivity, i.e.  $\varepsilon_{33}/\varepsilon_0$ ,  $\varepsilon_0$  is free space permittivity ( $8.854 \times 10^{-12} F/m$ ),  $r$  is the radius of the sample, and  $t$  is the sample thickness.

#### 3.5.1.1 Test Setup.

In order to facilitate testing, a National Instruments Analog Discovery 2 Data acquisition (DAQ) was used to measure and collect data. The test configuration

is shown in Figure 38 whereas the software used to record data is shown in Figure 39. The Analog Discovery 2 is a multi-function instrument to measure, visualize, generate, record, and control mixed signal circuits of all kinds. The primary features utilized in testing of this research were the two waveform generators and two channel multifunction oscilloscope.

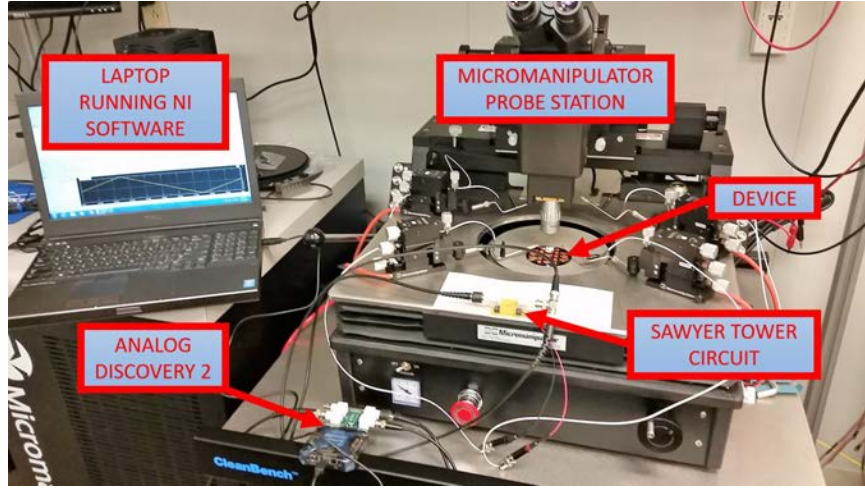


**Figure 38.** P-E hysteresis and characterization test setup

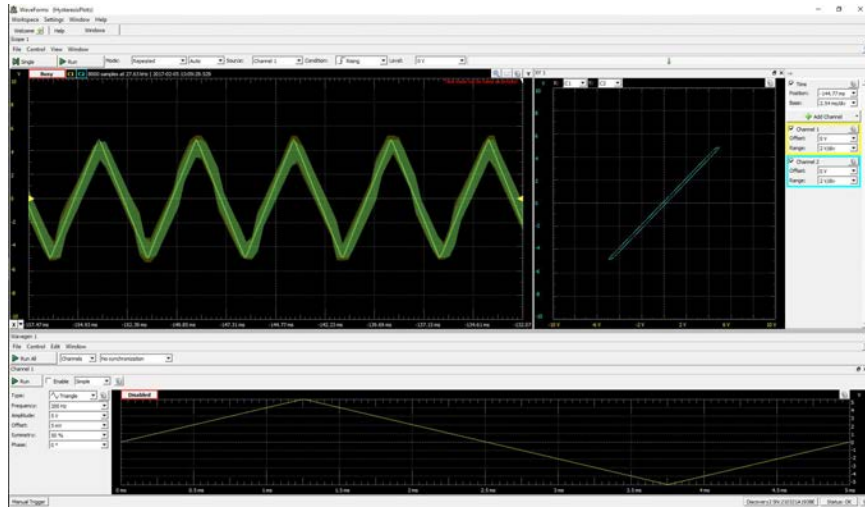


**Figure 39.** NI software (Digilent WaveForms) used with the Analog Discovery 2 for device characterization

In order to accurately measure the P-E hysteresis loop of the fabricated P(VDF-TrFE) devices, a custom circuit was designed and fabricated as shown in Figure 40

and 41. This circuit is then connected in series to the Analog Discovery 2 in order to obtain hysteresis measurements.

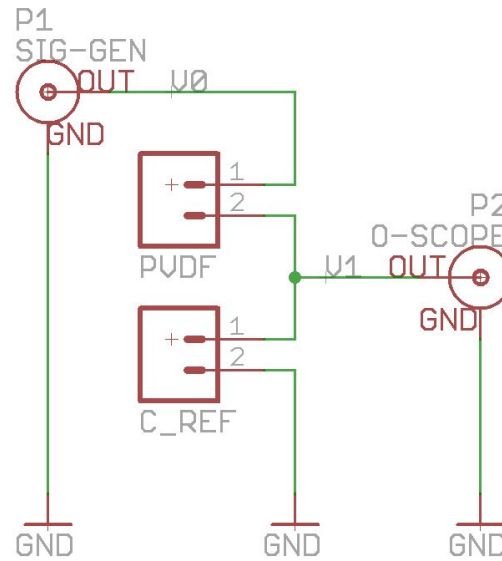


**Figure 40. Sawyer Tower Circuit Schematic**

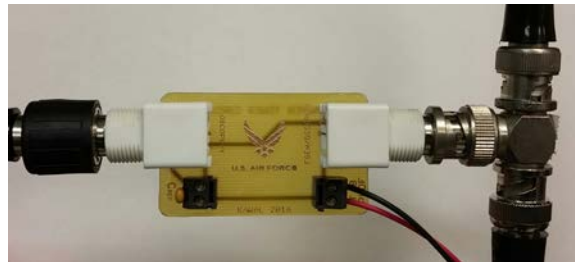
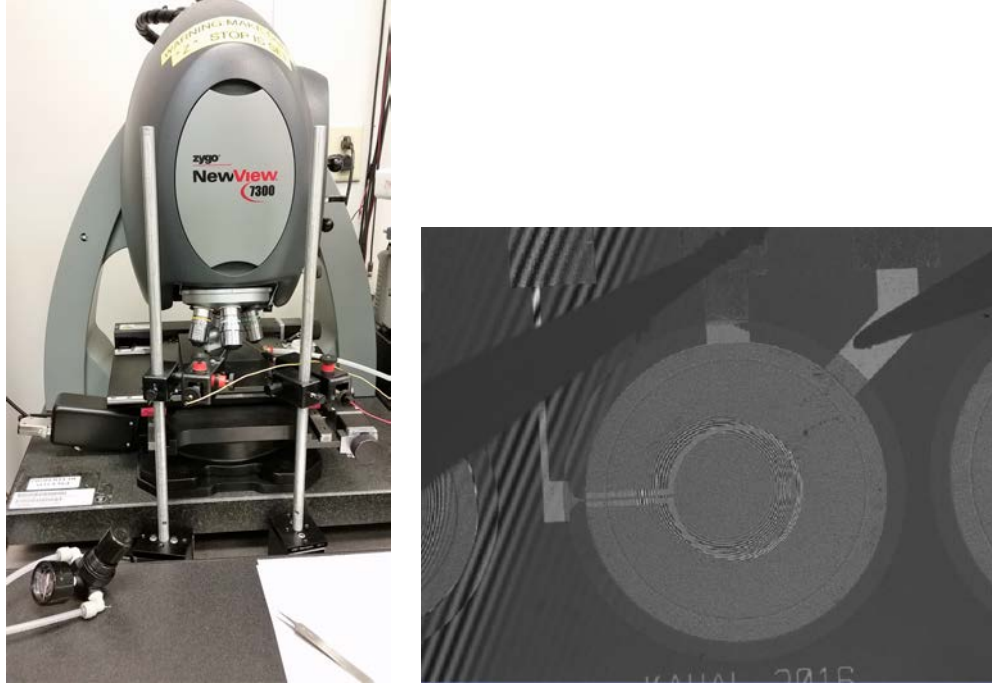


**Figure 41. Fabricated Sawyer Tower PCB used for measuring the ferroelectric properties of P(VDF-TrFE) - i.e. displacement polarity versus electric field**

### 3.5.2 Piezoelectric Membrane Characterization.

Under the optics of the ZYGO NewView™7300 probe tips were placed on the membrane probe pads and the grounding pads were connected to the input of an instrumentation amplifier and oscilloscope (Figure 42 & D-5).



(a) ZYGO NewView™ test configuration (b) SOI membrane device under the optics of the ZYGO NewView™

**Figure 42.** (a) The ZYGO NewView™7300 3D optical surface profiler and specialized air pressure platform was used to test the SOI membrane's stimulated response to changes in air pressure; (b) Probe tips were placed on the membrane probe pads under the optics of the surface profiler then connected to the input of an instrumentation amplifier and oscilloscope

The specialized platform shown in Figure 43 was used to test the electroactive membrane's stimulated response to changes in air pressure. The platform is a solid square slab of aluminum with a  $2mm$  hole drilled in the center of the horizontal surface. A second hole is drilled in to the side of the platform which intersects the hole from the horizontal surface. On the horizontal surface of the platform are two nylon clamps that are held in place by screws. These clamps keep the membrane device from drifting around due to the pressure on their under side as well as ensure a tight fit between the membrane DRIE cavity and the  $2mm$  hole. The output of a remotely controlled solenoid air valve is connected directly to the input of the threaded adapter which accepts a pressurized  $N_2$  line. An adjustable pressure regulator between the

$N_2$  source and the solenoid valve is used to control the pressure applied. The solenoid valve is electrically connected to the interface circuit shown in Figure D-7, which is subsequently connected to an NI Analog Discovery DAQ device in order to control the air pulse rate between 5-25Hz. This setup directs the pressurized  $N_2$  (2-15psi) to the back of the membrane at a specific interval, while the stimulated response is recorded via the oscilloscope datalogging feature of the NI Analog Discovery DAQ.

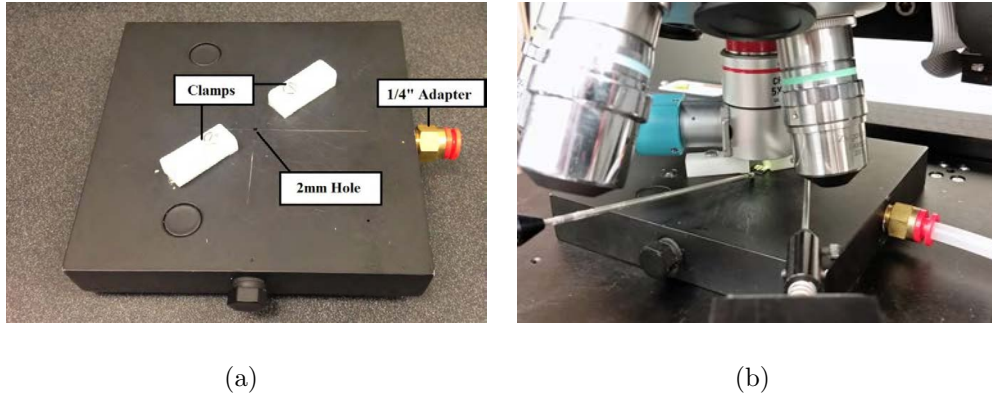


**Figure 43.** (a) specialized platform made from a solid slab of aluminum with drilled intersecting holes to direct pressurized  $N_2$  (2-15psi) to the back of the membrane devices - two nylon clamps are used to secure the membrane device during test; (b) platform holding samples in place while applying pressurized  $N_2$  to the back of the pressure sensor during deflection measurement with the ZYGO

[13]

### 3.6 Summary

This chapter detailed the design approach, modeling, device fabrication, post-fabrication processing, tools and equipment, and characterization and testing methodologies used in this research. The following chapters will present the data results collected during the evaluation of this unique design as well as the analysis of that data.

## IV. DATA

### 4.1 Chapter Overview

The previous chapter provides the result of processing and testing the behavior of electroactive polymer films. The primary challenge of these devices was the processing as it is directly related to overall electroactive performance of the device. The main processing steps involved deposition, patterning and poling. Once the processing was evaluated, the next step involved fabricating a suitable device in order to evaluate the energy harvesting capability on different structures to include a MEMS partial-bimorph structure. Chapter V will include a detailed analysis of the results presented here.

### 4.2 P(VDF-TrFE) Processing Results

The P(VDF-TrFE) fabrication process illustrated in Figure 35 was used throughout this research. Before fabricating began on any of the devices presented in this research, a deposition study was conducted in order to establish a process for depositing P(VDF-TrFE). Figure 44 shows the measured P(VDF-TrFE) film thickness based on the ratio of P(VDF-TrFE) to methyl ethyl ketone (MEK) based on the concentrations listed in Table 6.

**Table 6. P(VDF-TrFE)→MEK Concentration: % Weight/Volume**

| Weight/Volume concentration | P(VDF-TrFE) mass (solute) | Methyl Ethyl Ketone (MEK) volume (solution) |
|-----------------------------|---------------------------|---------------------------------------------|
| 8%                          | 2g                        | 25ml                                        |
| 10%                         | 2.5g                      |                                             |
| 15%                         | 3.75g                     | ↓                                           |
| 20%                         | 5g                        |                                             |

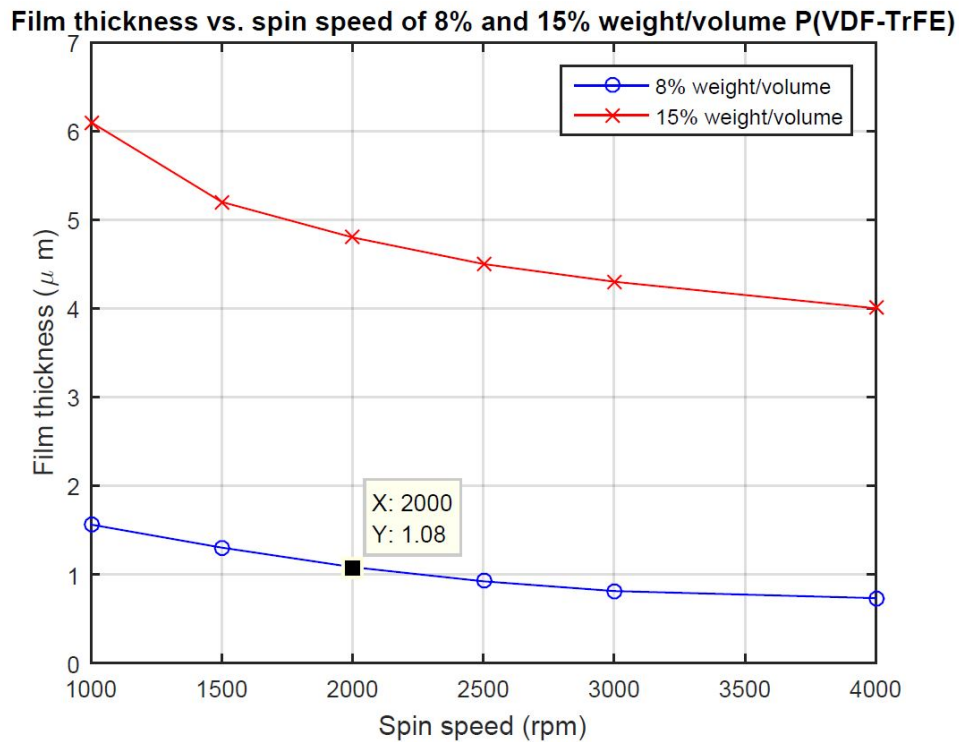


**Figure 44. Film thickness vs. spin speed with 8% and 15% weight/volume of PVDF-TrFE/MEK solution**

Thin film characterization was conducted using Filmetrics Model: F20-UV thickness measurement instrument. Figure 45 shows characterized film measurement recipe for pre-annealed P(VDF-TrFE), while Figure 46 shows the characterized film measurement recipe for annealed P(VDF-TrFE).

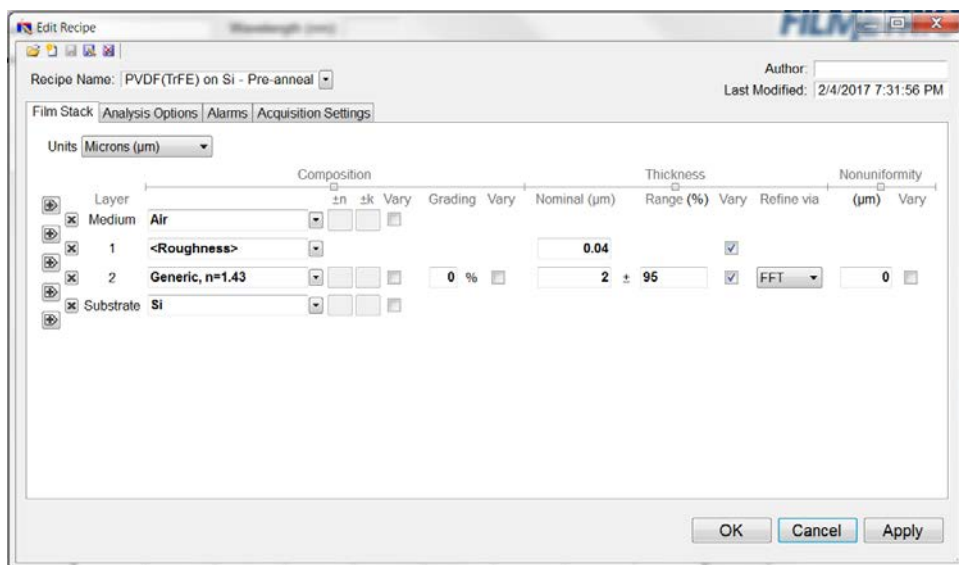


Figure 45. Filmetrics Model: F20-UV thickness measurement software pre-annealed P(VDF-TrFE) film thickness characterization recipe

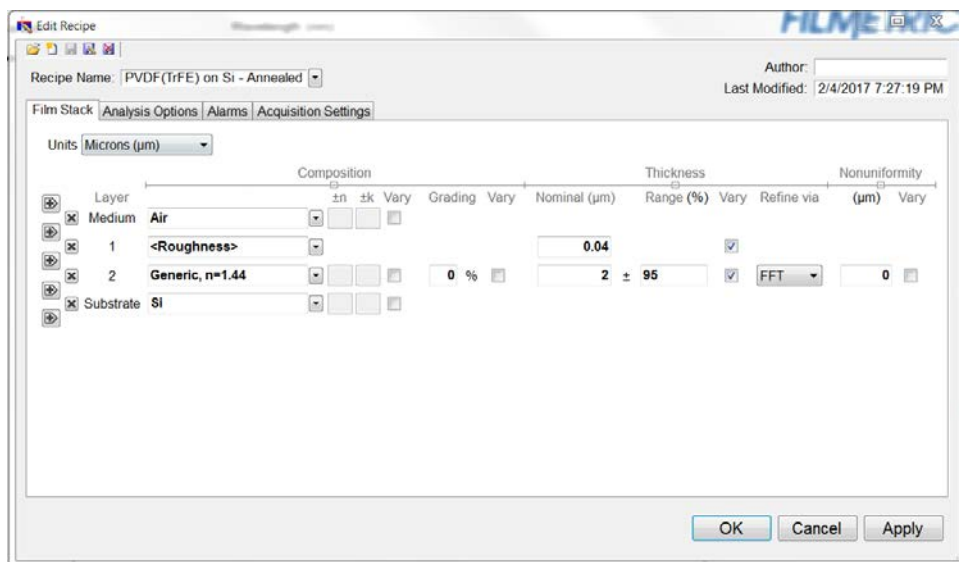


Figure 46. Filmetrics Model: F20-UV thickness measurement software post-annealed P(VDF-TrFE) film thickness characterization recipe

Wavelength reflectance spectra for both pre-annealed and annealed P(VDF-TrFE) thin films are shown in Figures 47 and 48.

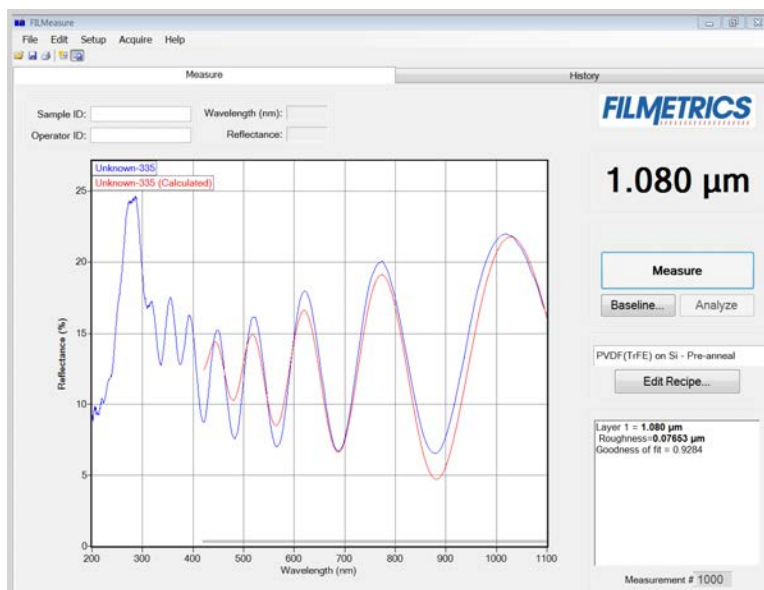


Figure 47. Filmetrics F20-UV measured film thickness reflectance spectra for pre-annealed 8% P(VDF-TrFE) solution spin coat at 2000RPM for 30 seconds ( $1.08\mu\text{m}$ )

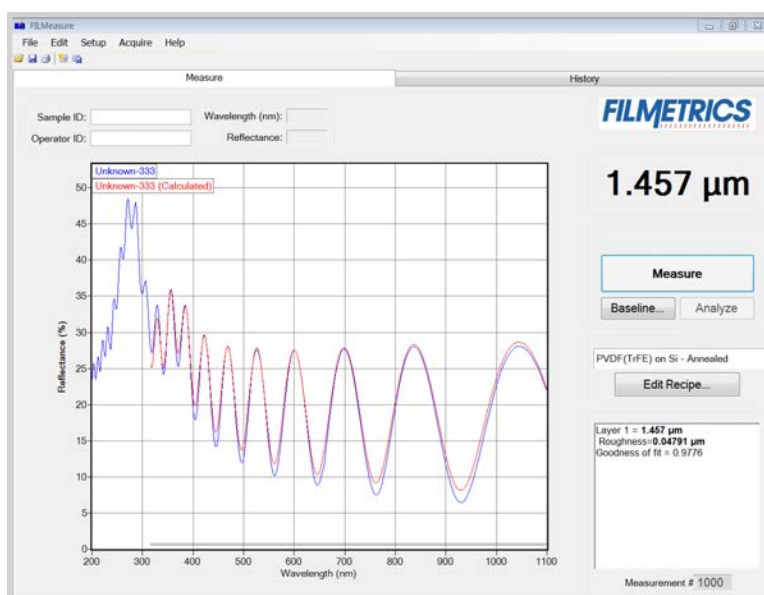


Figure 48. Filmetrics F20-UV measured film thickness reflectance spectra for annealed 8% P(VDF-TrFE) solution spin coat at 1500RPM for 30 seconds ( $1.46\mu\text{m}$ )

#### 4.2.1 Lithography and Patterning Results.

In order to pattern the P(VDF-TrFE) 1818 series as well as SU8 series photoresist was used as a Reactive Ion Etch (RIE) mask to pattern the P(VDF-TrFE) thin film.

However, before this could be accomplished, an etch study of 1818, SU8 and P(VDF-TrFE) was conducted in order to determine etch rates. A dry etch was performed under 75 SCCM  $O_2$  gas environment with 100 W RF power and 50mT pressure. Figure 49 shows the P(VDF-TrFE) etch rate was approximately  $234nm/min$ . Figures 50 and 51 show that 1818 series photoresist masks etch simultaneously with etch rates of  $154nm/min$ . Additionally, Figure 52 and 53 shows etch rates of  $210nm/min$  and  $235nm/min$  respectively.

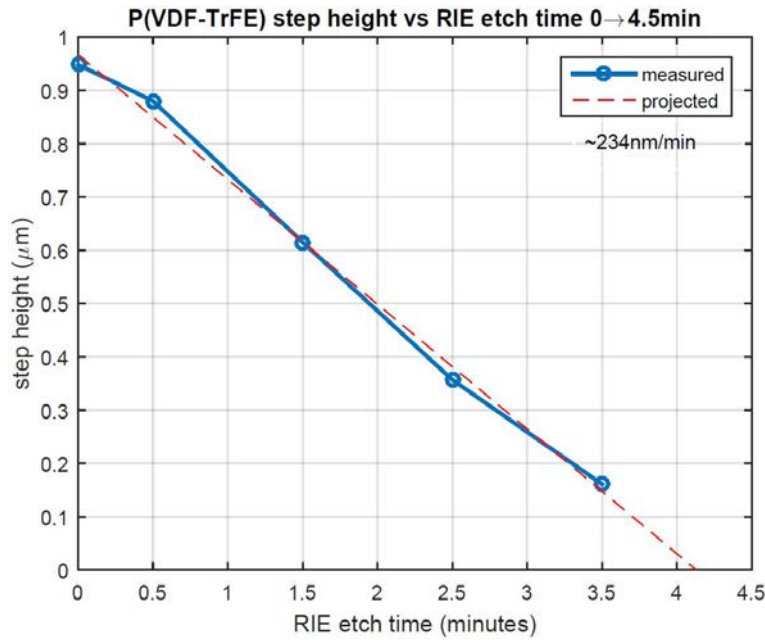


**Figure 49.** RIE etch study of un-patterned P(VDF-TrFE) on silicon substrate. Graph shows results from Filmetrics Model: F20-UV thickness measurement instrument ( $\pm 4\%$ )

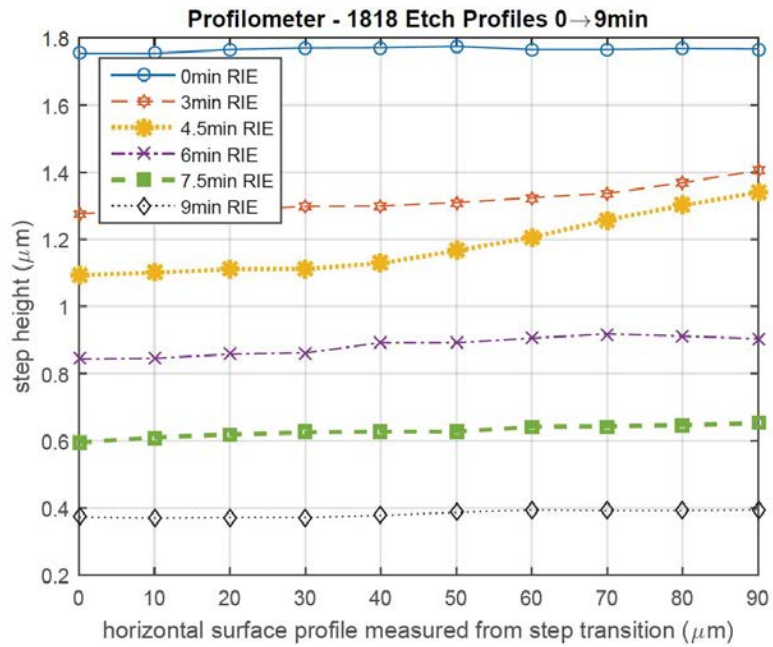


Figure 50. Measured profilometer surface plots of etched 1818 photoresist at various time intervals

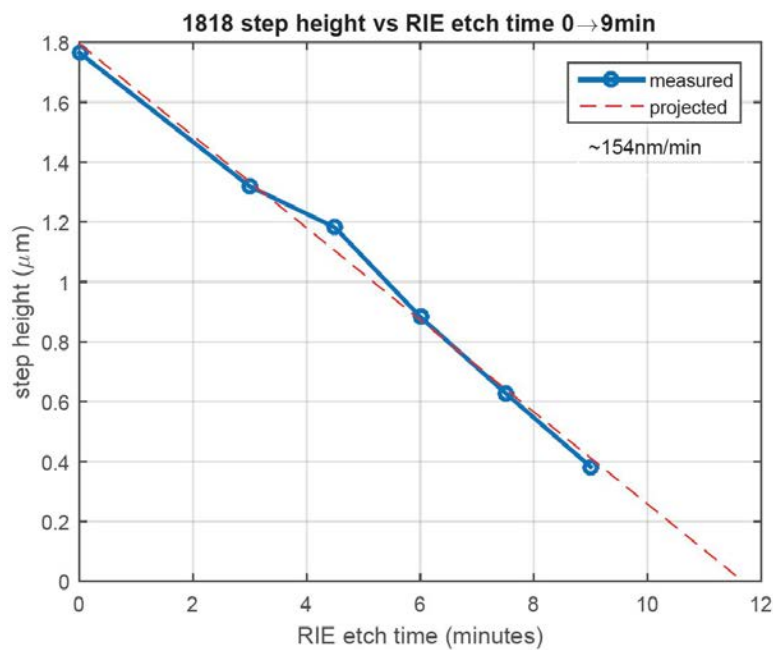


Figure 51. RIE etch study of patterned 1818 photoresist. Graph shows profilometer surface plots and respective etch durations

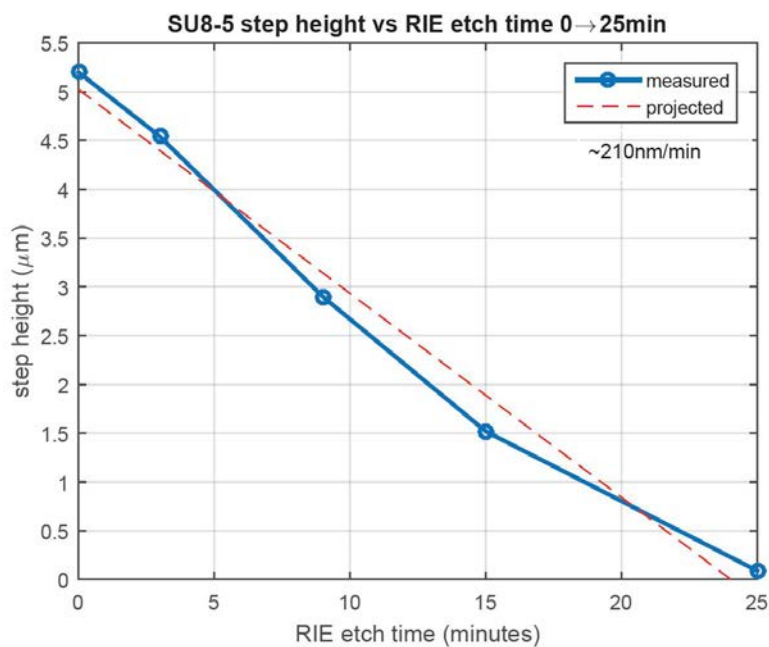


Figure 52. RIE etch study of patterned SU8-5 photoresist. Graph shows profilometer surface plots and respective etch durations

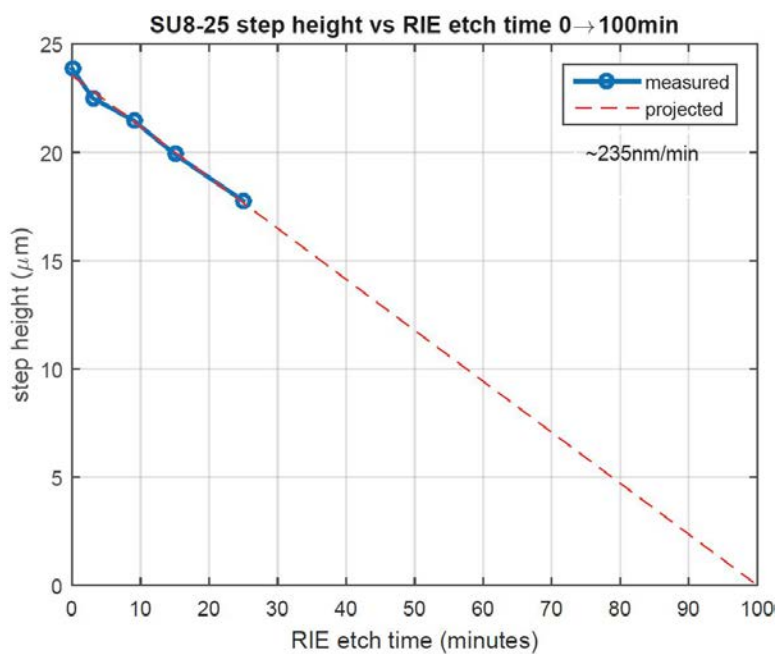


Figure 53. RIE etch study of patterned SU8-25 photoresist. Graph shows profilometer surface plots and respective etch durations

### 4.3 P(VDF-TrFE) Characterization and Beta Phase Measurements

The crystal structure of P(VDF-TrFE) plays an important role in how we can characterize it as a potential electro-active polymer for energy harvesting. Consequently the crystal structure is typically related to the composition of the co-polymer in addition to the annealing process. Recall in Chapter 2, whereas with the  $\beta$  crystalline phase of P(VDF-TrFE), the unit cell is orthorhombic, with each chain aligned and packed with the  $CF_2$  groups parallel to the b-axis as illustrated in Figure 19. Figure 54 shows the XRD pattern of P(VDF-TrFE) for different annealing temperatures (80, 90, 100, 110, 120, 140°C) in order to obtain information on the degree of the crystalline structure of the copolymer films.

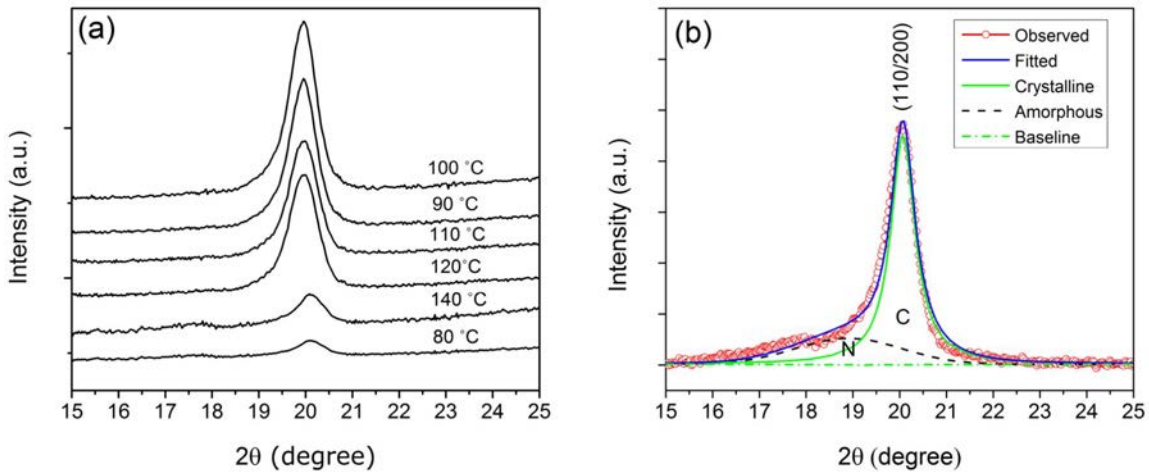


**Figure 54.** (a) XRD result of P(VDF-TrFE) thin films of different annealing temperatures (80, 90, 100, 110, 120, 140°C); (b) Deconvolution of the X-ray diagram for P(VDF-TrFE) thin film annealed at 100°C [1]

[41]

The degree of the crystalline structure,  $X_C$ , was evaluated from the ratio of area C to the total area under the diffraction curves  $N + C$  as seen in Figure 54. It is clearly seen in Figure 55 that annealing at 100°C induces the greatest increase in  $X_C$ .

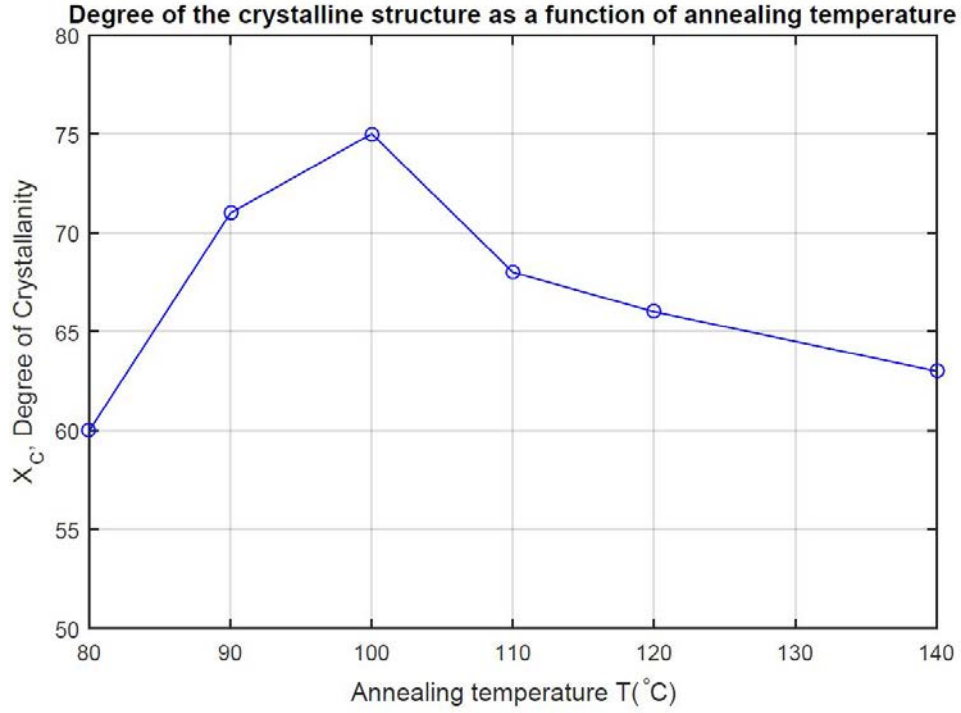


**Figure 55.** Dependence of the degree of the crystalline structure as a function of annealing temperature

Each phase of the P(VDF-TrFE) copolymer provides a characteristic FTIR spectrum. Due to the large mass of the fluorine atom, most infrared-active vibrations for P(VDF-TrFE) are concentrated between  $400 - 1500\text{cm}^{-1}$  [1]. Figure 56 illustrates the spectra of  $1\mu\text{m}$  thick P(VDF-TrFE) film annealed using differing methods. The three bands due to the  $\beta$  phase of P(VDF-TrFE) evaluated are  $850\text{cm}^{-1}$ ,  $1288\text{cm}^{-1}$  and  $1450\text{cm}^{-1}$  [42]. The intensity and the position of the absorption bands do not change much when annealed using different methods. This implies that the annealing method used does not affect the structure and  $\beta$  crystalline phase of P(VDF-TrFE).

### Raman Spectroscopy of P(VDF-TrFE) using different annealing techniques

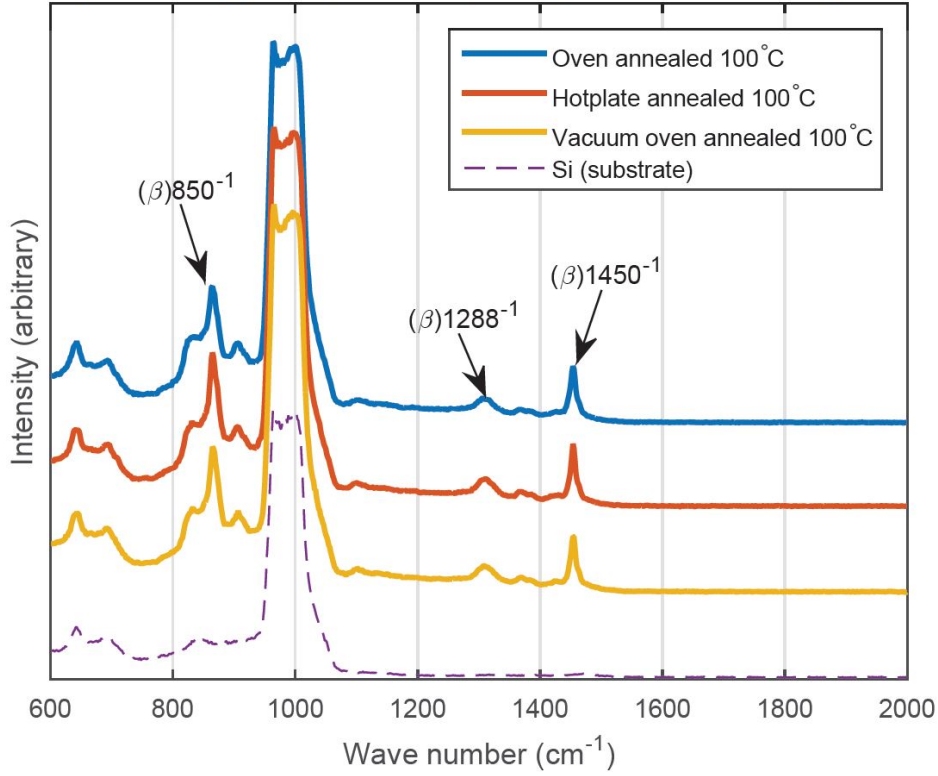


**Figure 56.** The strong absorption bands at  $850\text{cm}^{-1}$ ,  $1288\text{cm}^{-1}$  and  $1450\text{cm}^{-1}$  due to the  $\beta$  phase of P(VDF-TrFE) did not change much when different methods of annealing were used.

#### 4.4 EAP metal-insulator-metal device results

In the absence of an external electric field, ferroelectric materials possess an inherent, stable axis of polarization or electric dipole moment. This polarization can be changed by applying a strong electric field, on the order of MeV/m. However, with the thin film ferroelectrics presented here, only a few volts are required to change the polarization of the material. The EAP device configuration depicted in Figure 57 was tested resulting in a Polarization-Electric Field (P-E) hysteresis loop as illustrated in Figure 58.

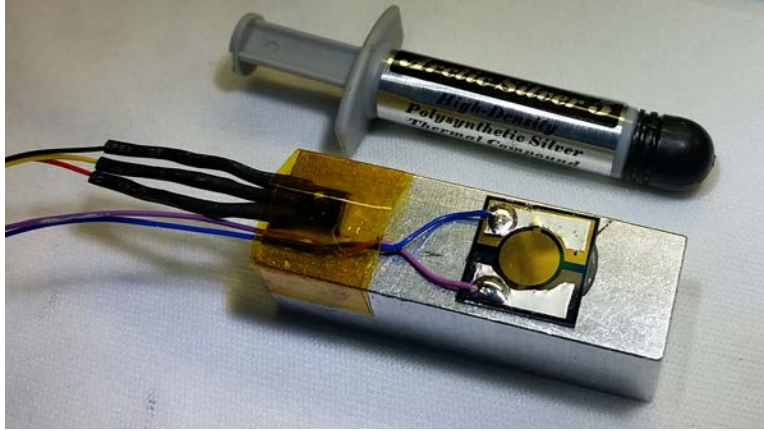


Figure 57. P(VDF-TrFE) metal-insulator-metal piezoelectric sample mounted to an aluminum thermal heat sink using a thermally conductive compound. Electrical connections were made using leads soldered to copper/tin tape containing conductive adhesive. In order to log temperature data, an analog temperature sensor (TMP36) with a measurement range of  $-40^{\circ}\text{C}$  to  $+125^{\circ}\text{C}$  ( $\pm 2^{\circ}\text{C}$ ) was mounted to the left of the device-under-test

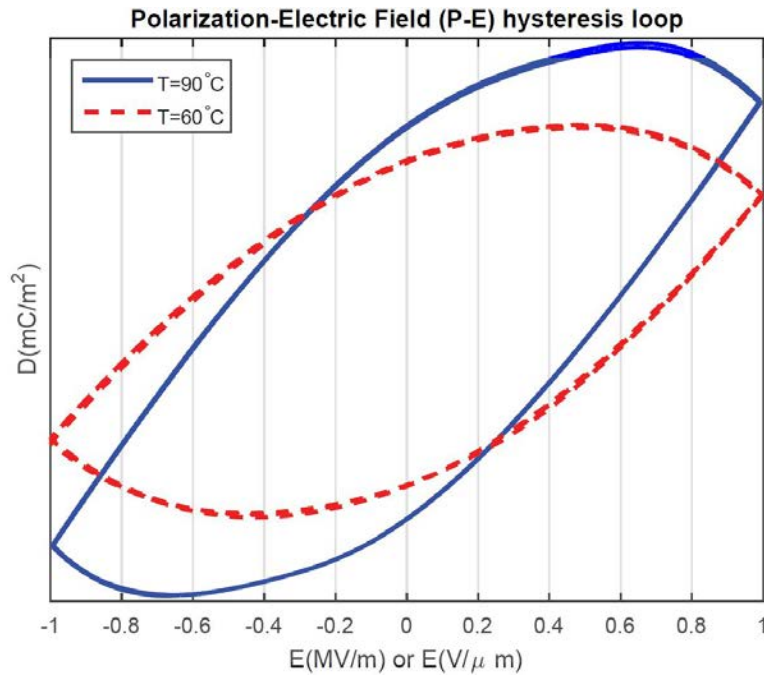


Figure 58. Measured Polarization-Electric Field (P-E) hysteresis loop of tested P(VDF-TrFE) metal-insulator-metal piezoelectric sample at  $60^{\circ}\text{C}$  and  $90^{\circ}\text{C}$

## 4.5 Membrane Device Results

For the membrane device shown in Figure 24 and 59, it is assumed that a large deflecting surface will yield greater energy production when applied for energy harvesting. Furthermore from prior research it is shown that when comparing the different sized membranes of the same thickness, it was found that a larger membrane resulted in a greater deflection over a given temperature range [6].

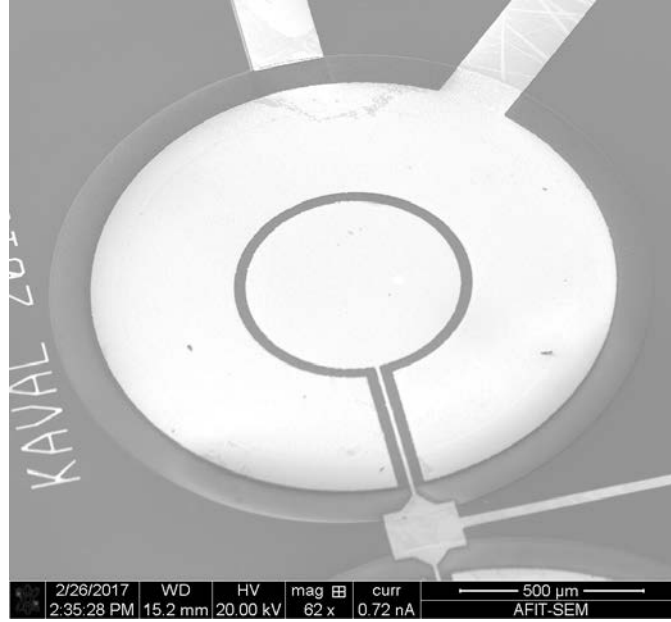


**Figure 59.** SEM image of fabricated SOI membrane P(PDV-TrFE) device structure with non-segmented bottom electrodes and segmented (inner/outer) top electrodes to capture energy response for both tensile and compressive modes as a result of membrane buckling post backside etch

Figure 60 and 61 show the buckling of a single SOI membrane device processed with P(PDV-TrFE). The buckling occurs once the handle is backside etched (DRIE). The relation between the membrane size and its deflection for the same processing steps is summarized in Table 7.

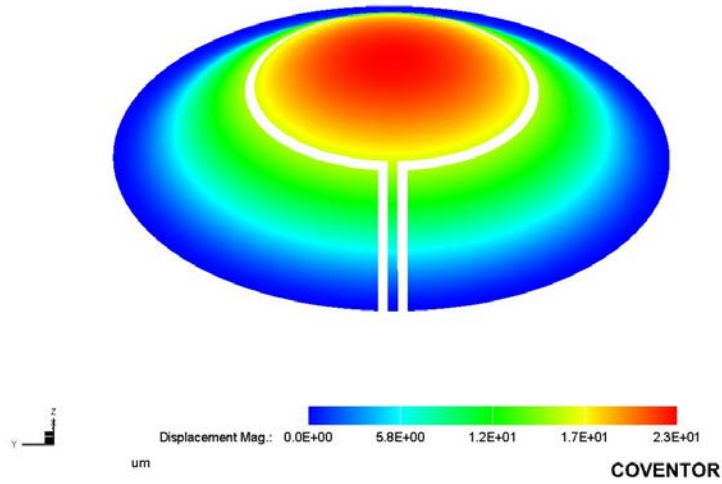


Figure 60. Simulated  $1250\mu\text{m}$  post-buckled membrane in its stable equilibrium position

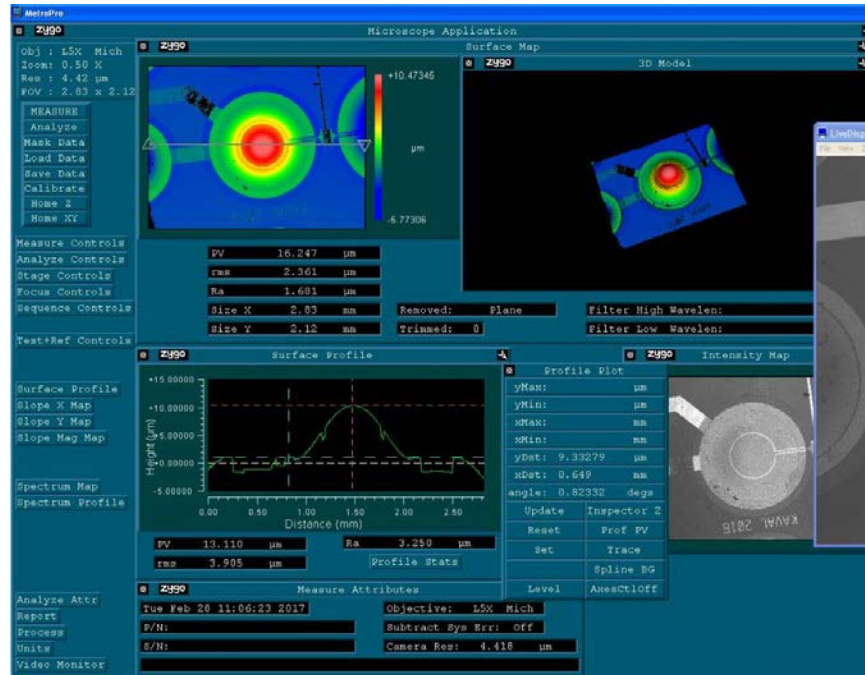


Figure 61. Screenshot of Zygo software measuring the deflection of a  $1250\mu\text{m}$  backside DRIE membrane device processed with P(PDV-TrFE). A top view image of the membrane is shown in upper left while a plot of the surface profile of the SOI membrane device is shown below

The SOI membrane device was electrically tested for continuity using the Micromanipulator probe station as depicted in Figure 62. This step was conducted to ensure that the electrodes weren't shorted due to voids in the P(VDF-TrFE) and/or

**Table 7. Table of membrane deflection results for FEM modeling and measured data for a membrane thickness of  $5\mu\text{m}$  and with an additional  $\sim 1.5\mu\text{m}$  of device process layers**

| Membrane Diameter ( $\mu\text{m}$ )   | 1,000 | 1,250 | 1,500 |
|---------------------------------------|-------|-------|-------|
| Measured Deflection ( $\mu\text{m}$ ) | 8.17  | 11.23 | 13.24 |
| FEM Deflection ( $\mu\text{m}$ )      | 12.26 | 15.40 | 19.21 |

by under/over wet metal-etching. The membrane devices were then mounted to a specialized platform under the optics of the ZYGO optical surface profiler, as shown in Figure 43 & 42, where deflection vs. voltage, deflection vs. pressure, and voltage vs. pressure measurements were collected.

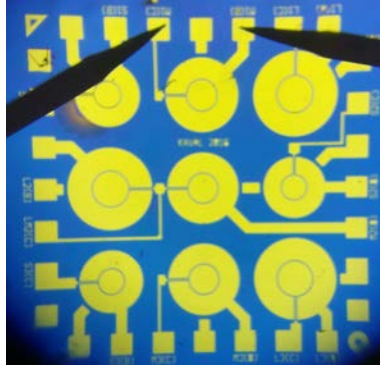


**Figure 62. Device probe pads viewed through the optical lens of the Micromanipulator probe station during electrical testing/data collection**

**Table 8. Membrane deflection vs. applied voltage**

| Applied Voltage | (diameter vs. deflection ( $\mu\text{m}$ )) |                     |                     |
|-----------------|---------------------------------------------|---------------------|---------------------|
|                 | 1,000 $\mu\text{m}$                         | 1,250 $\mu\text{m}$ | 1,500 $\mu\text{m}$ |
| 0V              | 8.177                                       | 12.470              | 13.241              |
| 1V              | 8.171                                       | 12.432              | 13.243              |
| 2V              | 8.155                                       | 12.415              | 13.247              |
| 3V              | 8.151                                       | 12.412              | 13.239              |
| 10V             | 8.143                                       | 12.410              | 13.237              |
| 25V             | 8.141                                       | 12.401              | 13.238              |

Voltage vs. pressure measurements were collected by using remotely controlled solenoid air valve with 10psi pressurized  $N_2$ . The solenoid valve was electrically connected to the interface circuit shown in Figure D-7 and connected to an NI Analog

**Table 9. Membrane deflection v.s applied pressure (1,250 $\mu$ m membrane)**

| Applied Pressure (psi) | Deflection ( $\mu$ m) |
|------------------------|-----------------------|
| 0                      | 12.53                 |
| 2                      | 12.95                 |
| 3                      | 13.59                 |
| 4                      | 14.24                 |
| 5                      | 15.22                 |
| 6                      | 15.59                 |
| 7                      | 16.62                 |
| 8                      | 16.82                 |
| 9                      | 16.97                 |
| 10                     | 17.83                 |

Discovery DAQ. Again, under the optics of the ZYGO NewView<sup>TM</sup>, probe tips were placed on the membrane probe pads which were connected to the input of an instrumentation amplifier (gain 10) and oscilloscope (Figure 42 & D-5). In order to obtain voltage response, the control the air was pulsed at 2-20Hz and directed to the back of the membrane. The stimulated response was recorded via the oscilloscope FFT analyzer function with the results listed in Table 10.

**Table 10. Membrane electrical response at (10psi) 2-20Hz pulsed N<sub>2</sub>**

| N <sub>2</sub> pulse rate (Hz) | dB Gain         |                 |
|--------------------------------|-----------------|-----------------|
|                                | Outer electrode | Inner electrode |
| 2                              | 50              | 55              |
| 5                              | 52              | 45              |
| 10                             | 45              | 35              |
| 20                             | 25              | 25              |

#### 4.6 PolyMUMPs Post-Fabrication Processing Results

Finite element modeling was conducted using CoventorWare©modeling software, to evaluate the addition of PVDF piezoelectric and top gold electrode film deposition post PolyMUMPS processing (prior to release). Partial-bimorph cantilever beam

material properties and geometries were evaluated as shown in Figure 63 and 64 and summarized in Table 11.

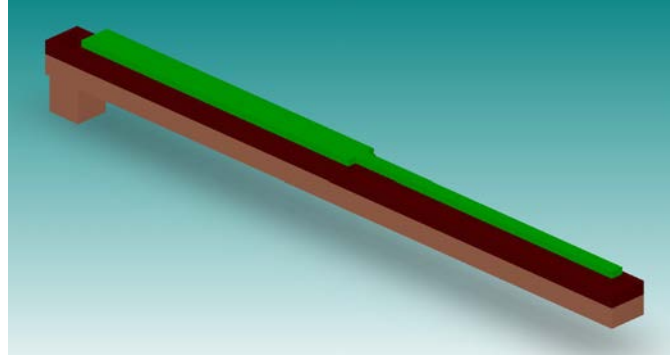


**Figure 63.** Cantilever, Modeled in CoventorWare©, with added Polymer and Top Electrode

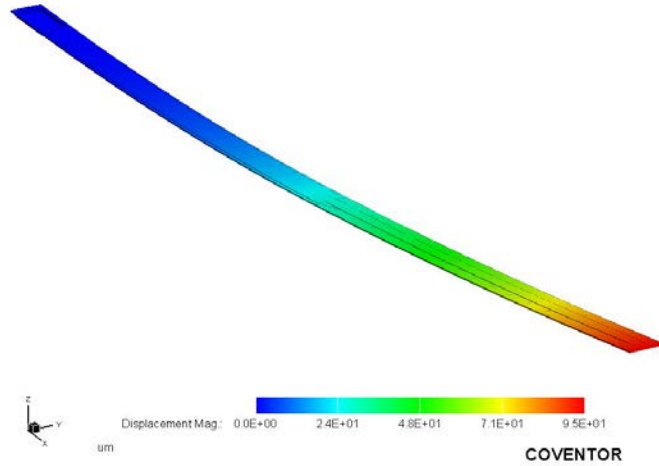


**Figure 64.** Modeled Initial Deflection of  $1000\mu m$  Cantilever ( $1.5\mu m$ )  
[43]

In simulation, the addition of PVDF and Gold reduced the deflection by 30% independent of actuator length. However, the residual stress given the added PVDF and metal layers was unknown and are considered to have a significant effect on initial deflections of the cantilever beams.

During the post-fabrication processing during this portion of this research P(VDF-TrFE) was deposited via spin coat method with a measured film thickness of  $1\mu m$ . Following a 5hr anneal at  $100^{\circ}C$  and 50mT, Cr( $30\text{\AA}$ ) and Au( $2000\text{\AA}$ ) were evaporated.

**Table 11. Results of Cantilever Deflection with/without Post Processing**

| <i>Length</i>           | Deflection ( $2\mu m$ )<br>( <i>Poly2, Au</i> ) | Deflection ( $3.5\mu m$ )<br>( <i>Poly2, Au, PVDF, Au</i> ) |
|-------------------------|-------------------------------------------------|-------------------------------------------------------------|
| $500\mu m \rightarrow$  | $24\mu m$                                       | $8.2\mu m$                                                  |
| $625\mu m \rightarrow$  | $37\mu m$                                       | $13\mu m$                                                   |
| $750\mu m \rightarrow$  | $54\mu m$                                       | $18\mu m$                                                   |
| $875\mu m \rightarrow$  | $73\mu m$                                       | $25\mu m$                                                   |
| $1000\mu m \rightarrow$ | $95\mu m$                                       | $32\mu m$                                                   |

As seen in previous research, devices post-processed with evaporated gold resulted in very little deflection [9]. It's suspected that this may have been due to the limited ability to control the chamber temperature, which then resulted in reduced residual stress over the bimorph segments. Once the metals were deposited, they were then patterned with 1818 photo-resist followed by a chemical wet etch. Once the metals were patterned as seen in Figure 65, the patterned metal features doubled as masking layers for the P(VDF-TrFE) during the RIE process to simultaneously dry etch the exposed P(VDF-TrFE) and any remove residual photo-resist used during the metal patterning.

Following the added processing steps, the post-processed MUMPs devices were released in HF. Figure 66 shows the completed device post HF release. Unfortunately, the initial release attempts were unsuccessful possibly due to residual photo-resist prior to release not fully removed during the RIE etch step.

## 4.7 Summary

In this chapter, P(VDF-TrFE) processing patterning and characterizations results were presented for the evaluation of several sensor design configurations introduced in this research. Evaluations of these designs were conducted using analytical modeling, finite element modeling, and experimental characterization and testing. Additionally, results from post-fabrication processing attempts were discussed.

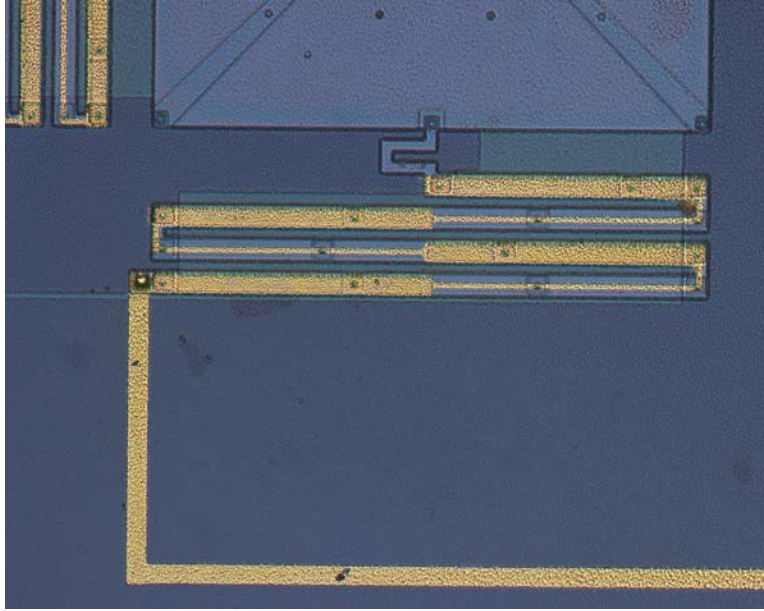


Figure 65. FEM post-release ('at-rest' state) of partial-bimorph structure with additional post processing steps to include deposition & patterning of P(VDF-TrFE) and metal electrode layers

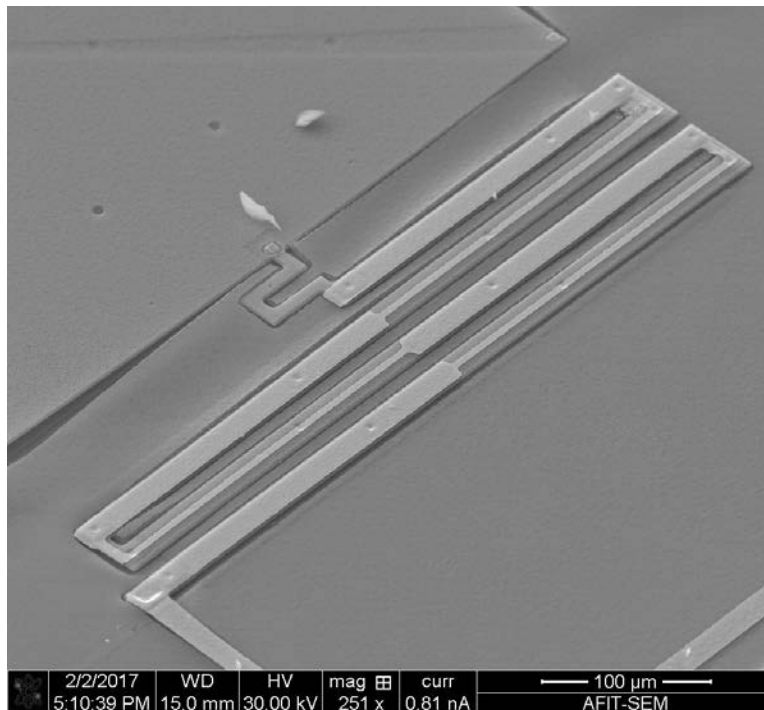


Figure 66. FEM post-release ('at-rest' state) of partial-bimorph structure with additional post processing steps to include deposition & patterning of P(VDF-TrFE) and metal electrode layers

## V. ANALYSIS

### 5.1 Chapter Overview

In this chapter, data collected during processing and testing the behavior of electroactive polymer films presented in the previous chapter is analyzed. Fabricated devices' operations are discussed, unusual or unexpected observations are explained, and hypotheses as to the causes are provided.

### 5.2 P(VDF-TrFE) Annealing

P(VDF-TrFE) (8% weight/volume) film cured in vacuum oven at 50mT and 100°C for 5 hours seen in Figure 67 demonstrated best adhesion and near  $\beta$ -phase formation due to the residual stress between the thin film and the substrate. Samples with P(VDF-TrFE) annealed in an oven or on a hot-plate showed poor adhesion on silicon substrate.

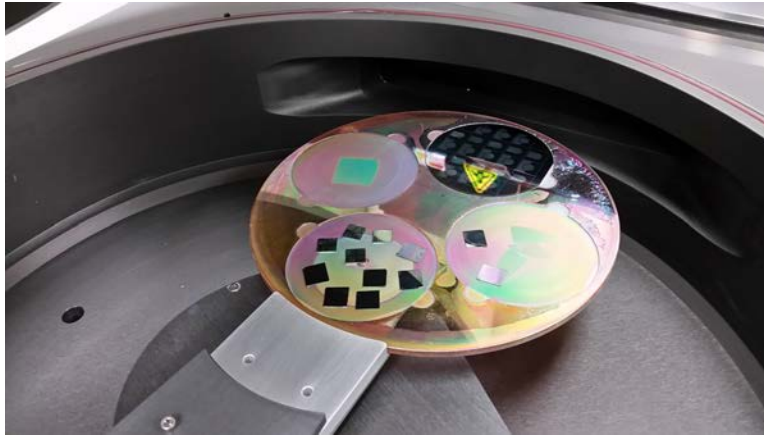
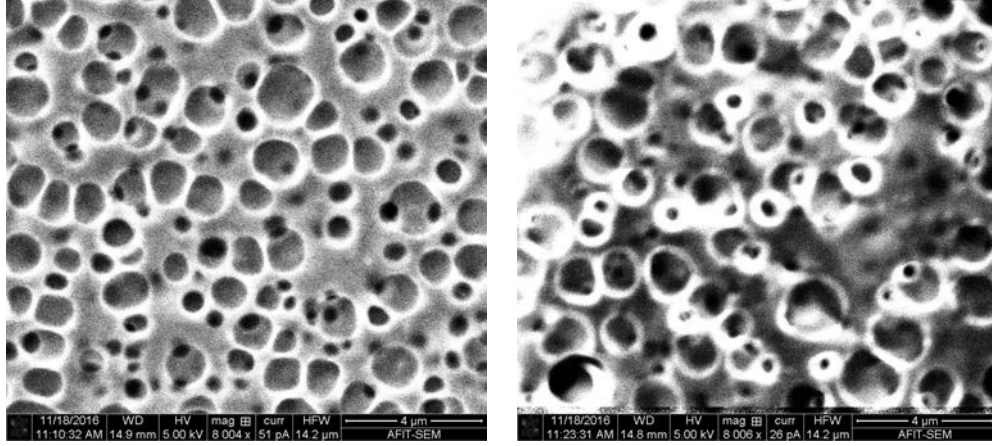


**Figure 67.** Samples prepared with P(VDF-TrFE) and loaded in PECVD chamber under 50mT vacuum for 5hr annealing at 100° followed by 6hr vacuum cool cycle

SEM images as seen in Figure 68 of vacuum oven versus hot-plate annealed samples were analyzed. The vacuum oven annealed samples showed a more uniform surface. However, both indicate a high porosity thin film.



(a) (vacuum oven) 50mT, 100°C for 5hrs

(b) (regular oven) 100°C for 5hrs

**Figure 68. SEM images of annealed P(VDF-TrFE) on silicon substrate**

### 5.2.1 P(VDF-TrFE) lithography and patterning.

Two types of photo-resist RIE masks were used to define PVDF patterns on a substrate. The first consisted of patterned 1818 photo-resist. The second consisted of using SU8. Both, however, presented challenges when patterned. Once the samples were annealed and now under elevated residual stress, the P(VDF-TrFE) immediately showed signs of swelling upon the deposition of 1818 photo-resist as seen in Figures 69 and 70. This occurred regardless of deposition technique.

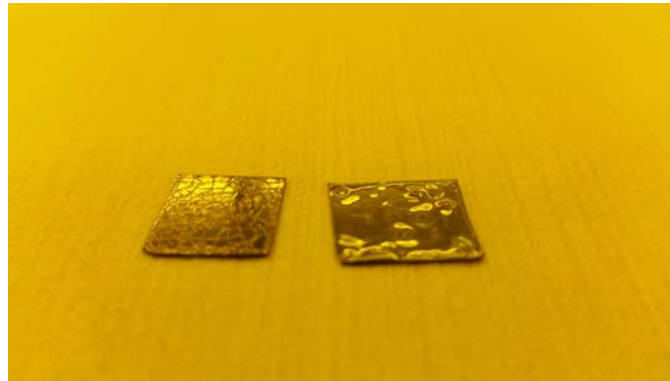


**Figure 69. Samples prepared with P(VDF-TrFE) and photo-resist deposited. Sample on left after 1818 photo-resist caused P(VDF-TrFE) to swell. Sample on right with SU8 did not show any signs of P(VDF-TrFE) swelling**

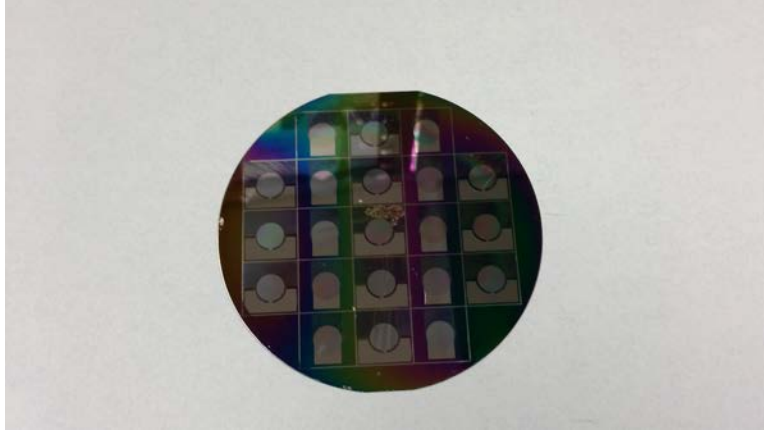
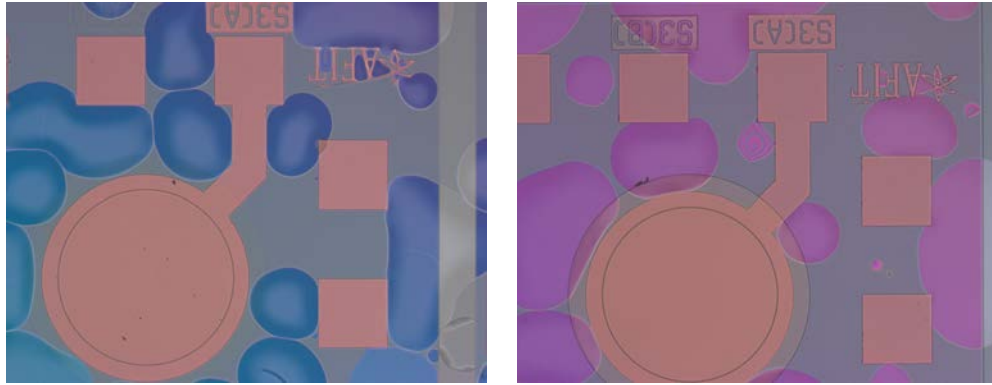


**Figure 70.** EAP device after 1818 photo-resist spin coat over 5hr annealed P(VDF-TrFE) at 100°. Note P(VDF-TrFE) swelling toward center of device

Additionally, during UV mask exposure, any P(VDF-TrFE) exposed to UV during lithography exposure de-laminated from any non-metal substrate as seen in Figures 71 and 72.



(a) Prior to 45sec 351-5:1 develop (b) After 45sec 351-5:1 develop (patterned 1818)

**Figure 71.** SOI membrane device processing post photo-resist (Shipley 1818) deposition and UV mask exposure (patterned) before-and-after 351 development

Unfortunately the use of 1818 could not be used because of the process compatibility. In fact, acetone, which is generally used to remove 1818, also dissolves P(VDF-TrFE). Therefore, SU8-5 was also spin coated 3000 rpm =  $5.2\mu\text{m}$  on top of the PVDF layer. After UV exposition and development, it was found that the

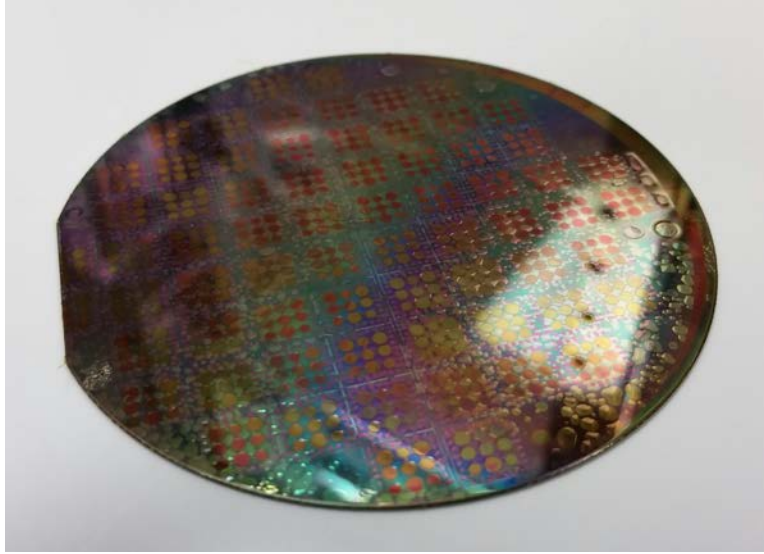


**Figure 72.** SOI membrane P(VDF-TrFE) lifting/bubbling in areas exposed to UV during patterning

SU8 developer also slightly dissolved the P(VDF-TrFE). Regardless, SU8 could be etched by RIE with an etching rate of approximately 200nm/min comparable to that for PVDF, making this process better suited for our purpose. Figure 73 shows a successfully patterned and RIE etched P(VDF-TrFE) device layer.

### **5.2.2 EAP and SOI device, P(VDF-TrFE) and top electrode patterning.**

Another challenge was the patterning of the top electrode. Three methods were explored. The first method shown in Figure 74 consisted of evaporating Ti/Au over patterned P(VDF-TrFE), followed by pattern and wet metal etch. This method proved difficult due to uncertainty of gold-etch on hand. The result was undercutting of masked lower electrode metals and consequently poor electrical contact traces and surfaces.

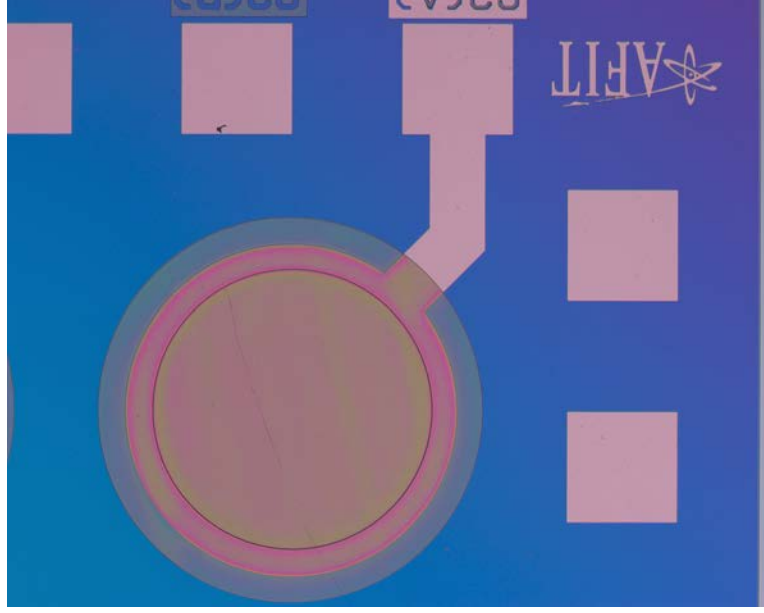
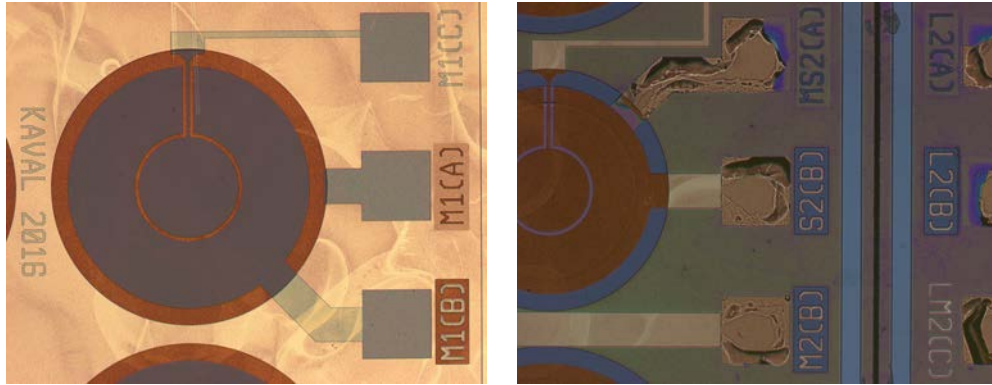


**Figure 73.** SOI membrane post dry etch process (RIE 100W-RF, 50mT and 75-SCCM  $O_2$ ). Etch rates were  $210nm/min(\pm 10nm/min)$  for photoresist (SU8-05) and  $234nm/min(\pm 10nm/min)$  for P(VDF-TrFE)

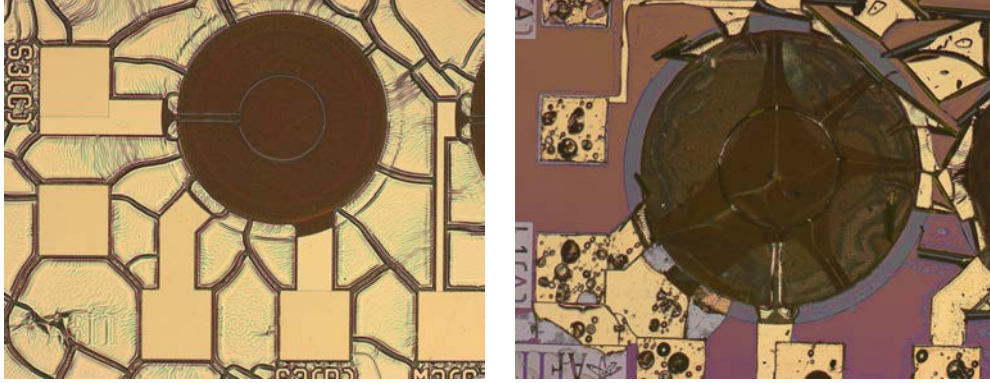


(a) Patterned 1818 photo-resist - pre wet metal etch (b) Undercutting after wet metal etch

**Figure 74.** Pattern and etch of evaporated  $250\text{\AA}/\text{Ti}$  and  $2000\text{\AA}/\text{Au}$  over P(VDF-TrFE). Note undercutting of top electrode pads after metal etch

The next method explored was a method of lift off where photo-resist was patterned over P(VDF-TrFE) before metal evaporation. However, since tape and/or acetone would also remove the P(VDF-TrFE), the patterned resist was flood exposed prior to metal deposition. By flood exposing prior to metal evaporation, the intent

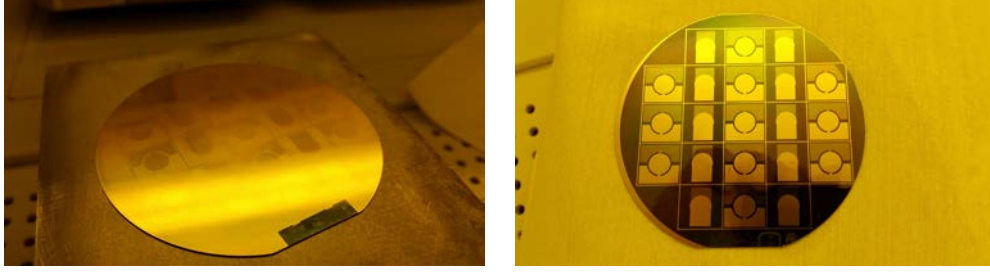
was to use the developer solution to lift off the metals as shown in Figure 75. Unfortunately this method was unsuccessful because the developer solution also removed the underlying P(VDF-TrFE).



(a) Metal evaporated over photo-resist (b) Attempted lift-off in 1:3 351 developer solution after 3min soak in ultrasonic bath prior to lift-off

**Figure 75. Failed lift-off attempt of evaporated Ti(250Å) and Au(2000Å) on top of patterned 1818 photo-resist and over P(VDF-TrFE) where everything lifted off**

The final and most successful method was by using the top metals as a masking layer. This was accomplished by evaporating metals over the un-patterned P(VDF-TrFE) then patterning and etching top metals, followed by a RIE dry etch. This method also avoided the incompatibility with the 1818 photo-resist and the SU8 developer solution. However, careful selection of materials/adhesion layers must be considered while patterning the top metals in order to prevent etching the bottom electrodes (due to the porosity of the P(VDF-TrFE)).



(a) Patterned top electrode prior to metal etch (b) After metal etch and P(VDF-TrFE) etch

**Figure 76. EAP top electrode pattern and etch, and using top electrode as P(VDF-TrFE) masking layer prior to RIE etch**

### 5.3 Piezoelectric Characterization

The hysteresis loop is one of the most important tools to characterize ferroelectrics. A significant amount of information can be extracted from the hysteresis loop. Charge-voltage schematics for some other common circuit elements are shown in Figure 77.

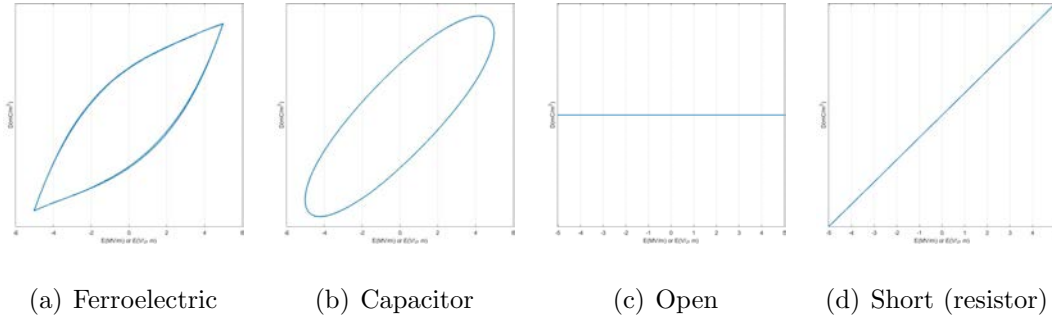


**Figure 77. Charge voltage response (in arbitrary units) of (a) ferroelectric, (b) capacitor, (c) open circuit, and (d) resistive element (or short circuit)**

Similar to other ferroelectrics, P(VDF-TrFE) copolymer exhibits remnant polarization. Figure 78 shows the hysteresis loops measured at 1 Hz with different applied voltages for the EAP device with P(VDF-TrFE) film thickness of approximately  $1\mu\text{m}$ .

As the temperature increases to 100°, the device starts to show greater hysteresis characteristics as seen in Figure 79.

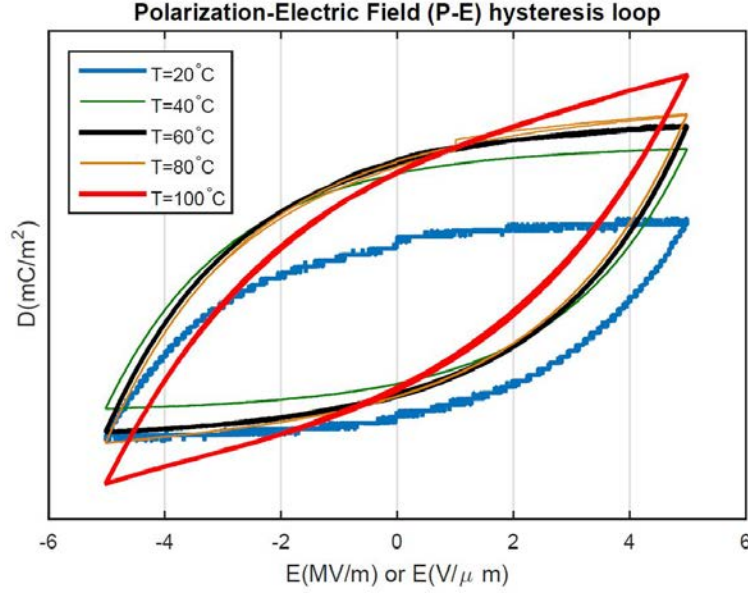


Figure 78. Hysteresis loops measured at 1 Hz with a fixed voltage and varying temperatures for the EAP device with P(VDF-TrFE) film thickness of approximately 1  $\mu$ m

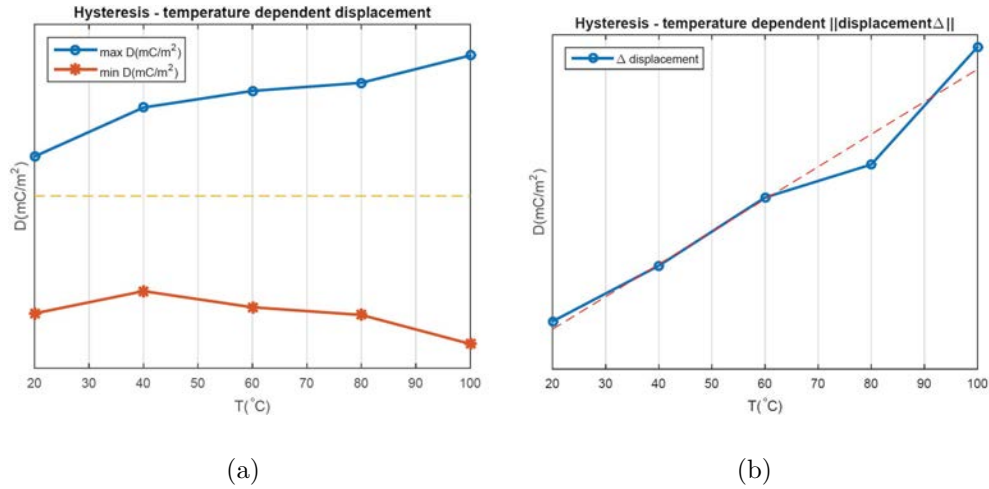


Figure 79. EAP hysteresis loop - (a) displacement temperature dependency and (b) (a) normalized displacement temperature dependency

## 5.4 SOI Membrane Results

The Membrane (metal-insulator-metal) energy-harvesting device structure with non-segmented bottom electrodes and segmented (inner/outer) top electrodes were designed to capitalize harvesting for both tensile and compressive modes of membrane buckling as shown in Figure 80 & 81

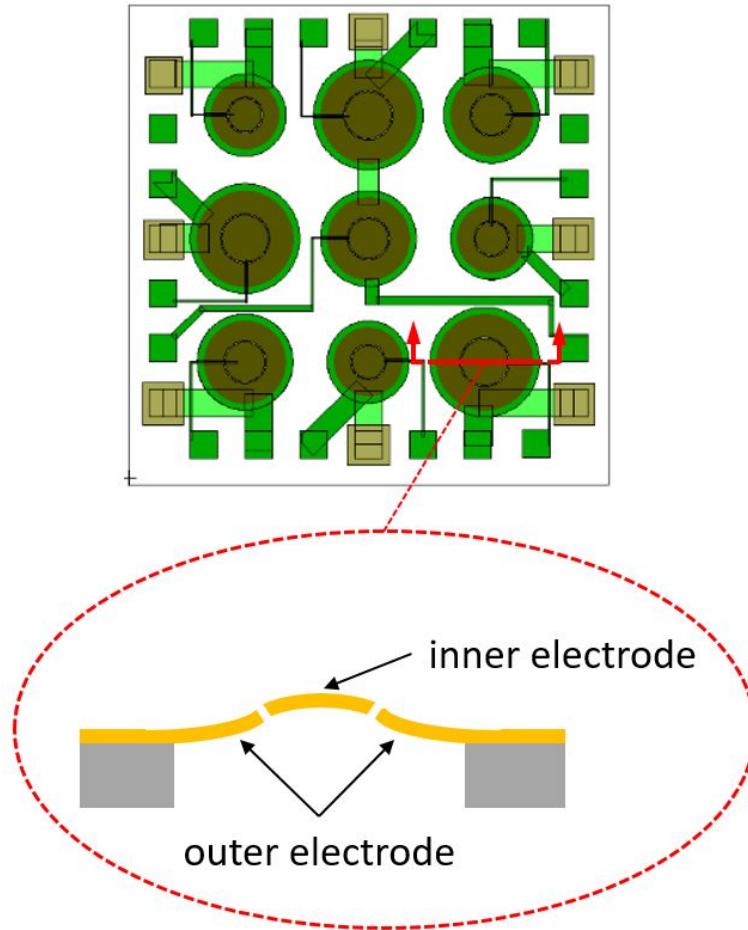
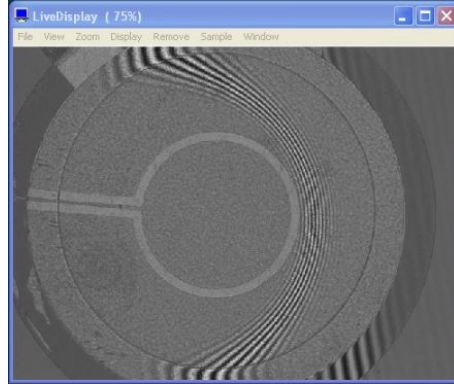
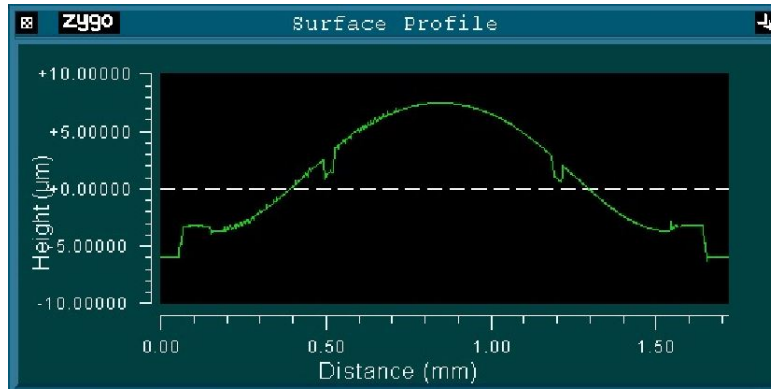


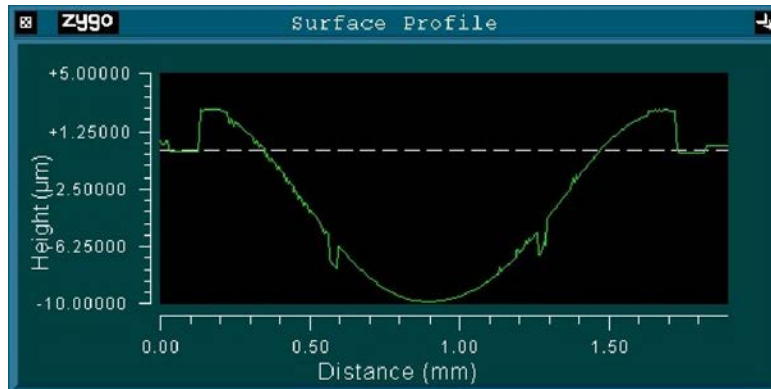
Figure 80. Segmented inner and outer electrodes for measured response in both tensile and compressive modes of membrane buckling



(a)



(b)



(c)

**Figure 81. (a) ZYGO surface profile scan of fabricated SOI device illustrating buckling modes of deflected membrane; (b) elevated buckling; (c) recessed buckling**

In order to measure the membrane electrical response to mechanical stimulation probe pads were connected to the input of an instrumentation amplifier at a gain of

10. The instrumentation amplifier was then connected to an oscilloscope in order to obtain the membrane voltage response to air pulsed at 2-20Hz. Since the response signal was very small due to the input impedance of the equipment ( $\sim 1\text{M}\Omega$ ) coupled with ambient electrical noise in the lab, the oscilloscope FFT analyzer was utilized to validate the measured response to air pressure independent of external sources (i.e. solenoid air valve). Based on the testing conducted, it was undetermined if a correlation exists between membrane deflection and the electrical response to each of the segments (i.e. inner & outer electrodes).

It was also noted after SOI device was released after the DRIE step, the P(VDF-TrFE) exhibited signs of thin-film cracking as seen in Figure 82. This may have contributed to the weak electrical response to mechanical stimulation as presented in Chapter 4.

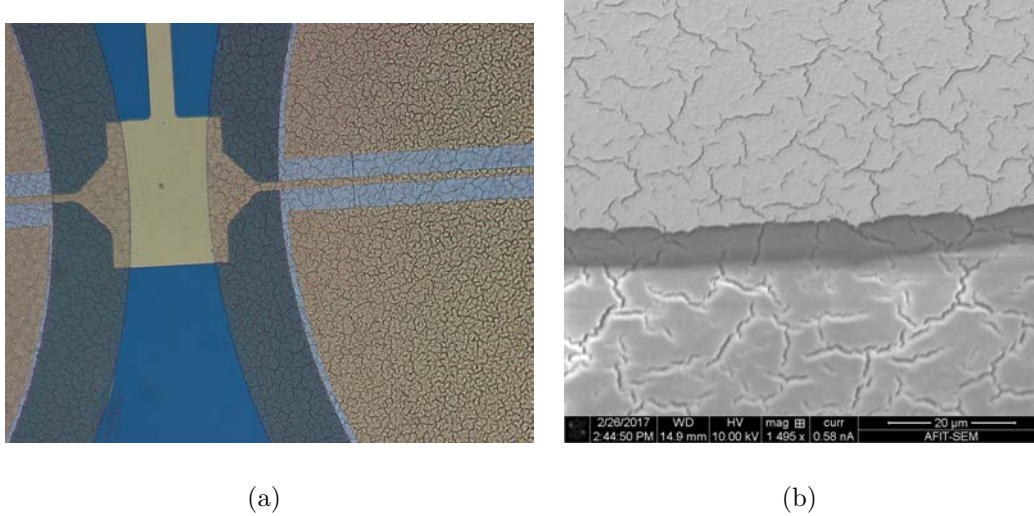


**Figure 82.** (a) optical and (b) SEM images of SOI membrane device indicate P(VDF-TrFE) thin film cracking after DRIE

## 5.5 PolyMUMPs Processing Results

Due to the issues with receiving correct PolyMUMPs devices, all device characterization and testing was performed on a limited number of devices provided (remnants

of previous PolyMUMPs fabrication runs). However, this did not hinder the techniques used for the post processing.

Due to the small size of the PolyMUMPs devices, they were first bonded to a 3" carrier wafer using a small amount of 1818 photo-resist as seen in Figure 83. However, the use of 1818 caused issues during release, because the residual 1818 could not be removed with acetone. However, methanol could be used but was proven to be less effective with 1818 photo-resist removal. Consequently, prior to HF release I believe trace amounts of photo-resist may have prevented full release of the device.

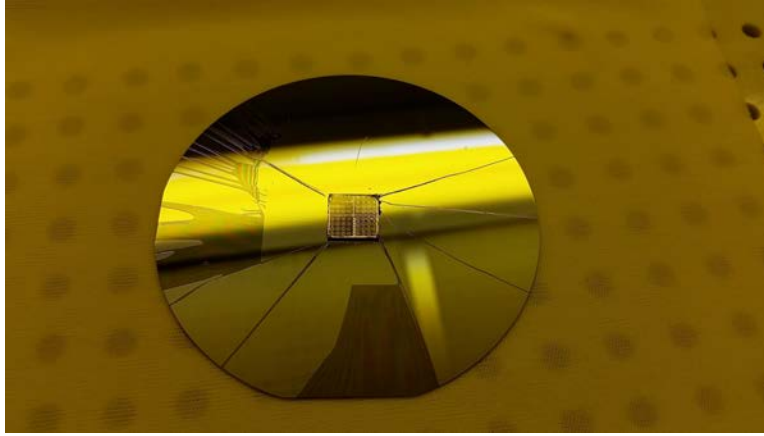
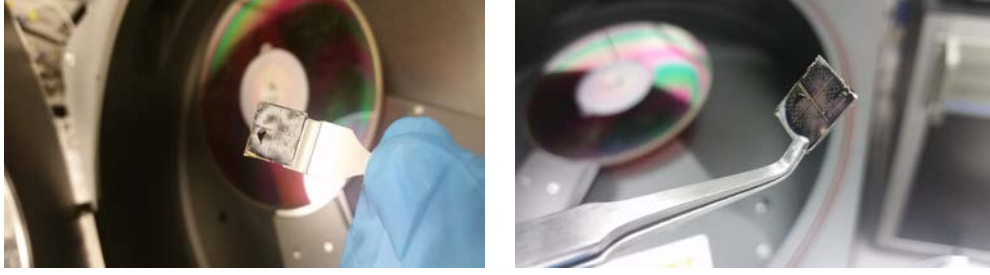


**Figure 83. PolyMUMPs device mounted to 3" carrier wafer for ease of patterning. Later revealed to be an unnecessary step**

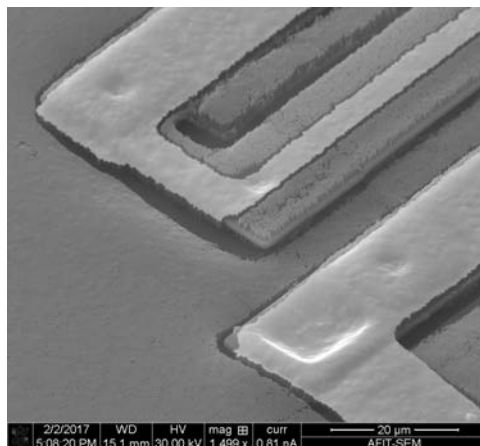
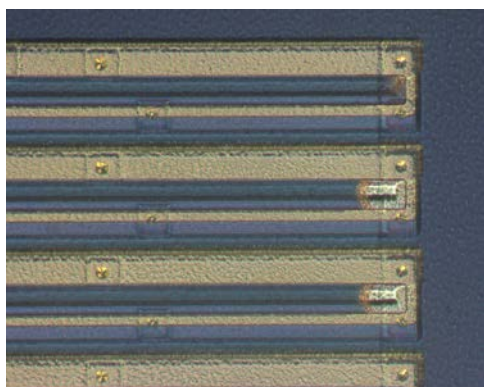
The process used for patterning the polyMUMPs consisted of first spinning on a  $1\mu\text{m}$  of P(VDF-TrFE), followed by a 5hr anneal at  $100^\circ$  and 50mT, then evaporating Cr( $30\text{\AA}$ ) and Au( $2000\text{\AA}$ ) followed by pattern and two etch steps (metal etch and RIE). During the RIE etch step for removing the exposed P(VDF-TrFE) it was noted that etching began to slow drastically as seen in Figure 84. It was later discovered that the Minilock-Phantom reactive ion etcher (RIE) machine being used was not properly stabilizing the reflected energy and therefore not sustaining the plasma required for a proper etch. Once this issue had been resolved, a close eye was kept on the measured etch parameters in order to confirm proper etch rates.



(a) Failed RIE attempt ( 58W reflected RF)    (b) Resulting etch (1-2W reflected RF)

**Figure 84. PolyMUMPs post processing P(VDF-TrFE) RIE etch - 234nm/min at 75W(RF) 50mT and 100 SCCM(O<sub>2</sub>)**

Once the devices were patterned and etched, they were submerged in HF for 15min. Upon the 15min soak in HF, the devices were gradually and carefully soaked in several methanol baths then dried in a carbon dioxide (CO<sub>2</sub>) dryer. Figure 85 shows the devices before and after the release step. As seen in Figure 86 despite best efforts to achieve a fully released post-processed device, this portion of the research was only partially successful in that the P(VDF-TrFE) remained intact throughout the entire process. There were two main contributing factors which hindered full release of the devices. First, the lack of devices on hand, and second, the result of the residual protective photo-resist remaining on the device prior to the HF soak.



(a) Post-processed top electrode prior to HF release (b) SEM image of post-processed top electrode prior to HF release

**Figure 85. PolyMUMPs top electrode Au/P(VDF-TrFE) patterned and etched, before and after HF release**

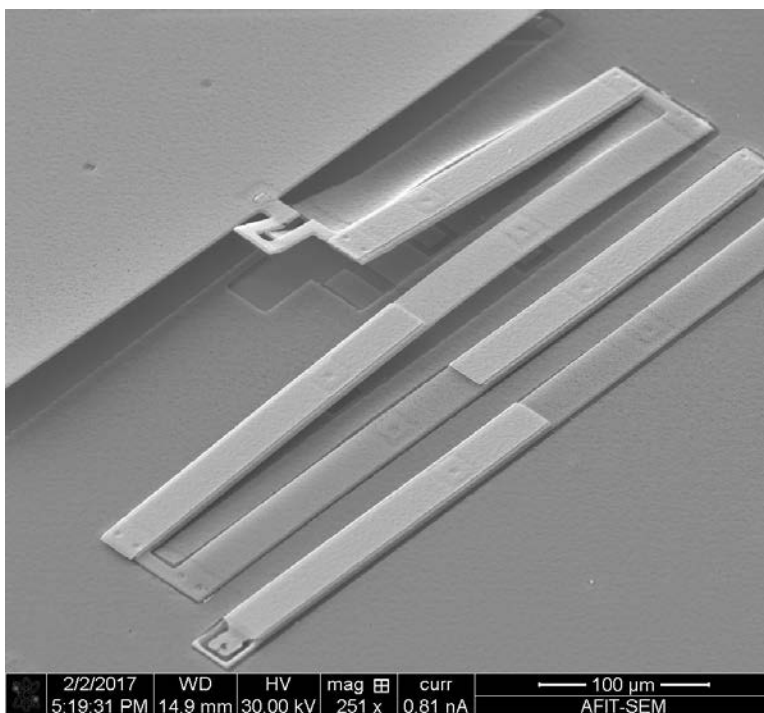


**Figure 86. SEM image of partially-released PolyMUMPs device**

## 5.6 Summary

This chapter presented analysis of the data collected during the processing and evaluation of several P(VDF-TrFE) enabled designs, fabricated device operations, and post-fabrication results were discussed. Observations were explained and suspected causes for all observations were provided. Conclusions found during this research, the significance of this research, and recommendations for future research are provided in the following chapter.

## VI. CONCLUSIONS AND RECOMMENDATIONS

### 6.1 Chapter Overview

This chapter provides a summary of the research conducted on the study of P(VDF-TrFE) for mechanical-to-electrical energy-harvesting design, the significance of the research, and recommendations for future research on this topic. Following this, recommendations of future work will be provided based on both observations of what could be improved with the current design, as well as new ideas with regard to energy harvesting devices.

### 6.2 Conclusions of Research

The work presented in this thesis provided the framework for several unique approaches in order to characterize P(VDF-TrFE) for energy-harvesting application. The first device was intended to be used for the characterization and processing involved in fabricating a P(VDF-TrFE) based energy-harvesting assembly. Multiple avenues of fabrication and processing were conducted which resulted in the validation of viable processing options toward the implementation of a full scale energy-harvesting array capable of producing usable amounts of power.

The processing and characterization provided a foundation to evaluate the implementation of several designs. These designs included an SOI device as well as a PolyMUMPs MEMs device to determine the required processing steps compatible with such devices. Additionally, it showed that there are specific processing requirements necessary for the production of an optimal piezoelectric response from P(VDF-TrFE).

In regard to the PolyMUMPs post processing, finite element modeling partially validated the initial deflections of a partial-bimorph cantilever beam. It demonstrated

the feasibility of using P(VDF-TrFE) for potential sensing application for agile laser beam-steering micro-mirror array applications, currently of interest to the U.S. Air Force.

### **6.3 Significance of Research**

The goal of this research was to design, model, fabricate, and test/characterize a high fill-factor, large aperture, out-of-plane, sensor/actuation assembly capable of generating electrical charge with an eventual project goal of collecting and storing usable energy. Therefore since the harvester performance is proportional to resonant frequency, a known proof-mass was used to establish the dominant resonant frequency. Other applications of this research include off-grid remote sensing applications as well as a multitude of commercial systems requiring a sustained sources of power. Ultimately, knowledge gained from this research will increase the efficiency of consumable power sources.

### **6.4 Recommendations for Future Research**

Unfortunately, due to the time and fabrication constraints of this research, full characterization of the PolyMUMPs designs could not be accomplished. While a majority of the stated goals for this research were met, further research is needed to optimize the mechanical to energy harvesting ability of these devices.

For this reason, the following recommendations for future work are presented based on knowledge gained during this research: examining the effects of using different polar solvent solutions to dissolve P(VDF-TrFE); determining the cause of the P(VDF-TrFE) thin-film cracking after DRIE processing (application thicker electro-active polymer films or encapsulation); characterizing the effects of varying the poling voltage and duration on the P(VDF-TrFE); further characterizing the piezoelec-

tric response when applied to cantilever beam structures; investigating the effect of P(VDF-TrFE) structure geometry on harvested power, specifically surface area and film thickness; and examining the long-term durability of these devices under typical and out-of-spec usage and environmental exposure, particular to Uv light.

## **6.5 Summary**

This chapter concluded this research by highlighting the conclusions and the significance of this research, and providing recommendations for future research topics.

# Appendix A.

## A-1 P(VDF-TrFE) Membrane Fabrication Process Follower

| Wafer<br>SOI | Purpose<br>SOI - MEMBRANE                                                                                                                                                                                                                                                                                                                                                                                                                                | ID<br>SOI | Mask | Process<br>Membrane Device | Print Date<br>11/15/2016                   |              |
|--------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------|------|----------------------------|--------------------------------------------|--------------|
| Init.        | Process Step                                                                                                                                                                                                                                                                                                                                                                                                                                             |           |      |                            | Notes                                      | Date<br>Time |
|              | <b>I. Solvent Clean (by hand)</b> <ul style="list-style-type: none"> <li>• Turn on hot plate and set to 110°C</li> <li>• 30 sec acetone rinse</li> <li>• 30 sec methanol rinse</li> <li>• 30 sec DI water rinse</li> <li>• Dry with nitrogen on texwipe</li> <li>• 2 min 110°C hot plate bake</li> <li>• Turn off hot plate*</li> </ul>                                                                                                                  |           |      |                            | <b>Start Date</b><br><br><b>Start Time</b> |              |
|              | <b>II. PECVD SiO2 Deposition</b> <ul style="list-style-type: none"> <li>• Deposit 200-300nm of SiO2 to serve as bottom electrical isolation layer</li> </ul>                                                                                                                                                                                                                                                                                             |           |      |                            |                                            |              |
|              | <b>III. Photoresist Application (bottom Oxide)</b> <ul style="list-style-type: none"> <li>• Photoresist(1818):</li> <li>• Turn on hot plate and set to 110°C*</li> <li>• Place wafer on spinner (polished side up)</li> <li>• Flood with 1818 photoresist</li> <li>• 30 sec spin at 4,000 rpm</li> <li>• 1min @ 110°C hot plate bake</li> <li>• Turn off hot plate*</li> </ul>                                                                           |           |      |                            |                                            |              |
|              | <b>IV. Expose 1818 – "Mask 0: Oxide"</b> <ul style="list-style-type: none"> <li>• Start EVG select 1818 process tab</li> <li>• Clean contact mask under solvent hood</li> <li>• Follow steps to center wafer/align to mask</li> <li>• Expose using EVG aligner (7 sec)</li> </ul>                                                                                                                                                                        |           |      |                            |                                            |              |
|              | <b>V. Develop 1818</b> <ul style="list-style-type: none"> <li>• Place wafer on base hood spinner</li> <li>• 45sec develop with 351:DI (1:5) at 500rpm (start-stop method)</li> <li>• 30 sec DI rinse at 500rpm</li> <li>• Dry with nitrogen at 500rpm.</li> <li>• Dry on tex wipe with nitrogen</li> <li>• Verify develop under microscope, repeat this block if necessary</li> <li>• Record thickness of the patterned 1818 on profilometer.</li> </ul> |           |      |                            |                                            |              |
|              | <b>VI. Etch Oxide</b> <ul style="list-style-type: none"> <li>• Partially fill small container with BOE. (enough to cover sample).</li> <li>• Submerge sample in BOE for 1 minute. Slight agitation.</li> <li>• 30 second rinse and dry with nitrogen.</li> </ul>                                                                                                                                                                                         |           |      |                            |                                            |              |
|              | <b>VII. Photoresist Application</b> <ul style="list-style-type: none"> <li>• Photoresist(1818- bottom electrode patterning &amp; lift-off):</li> <li>• Turn on hot plate and set to 110°C*</li> <li>• Place wafer on spinner (polished side up)</li> <li>• Flood with 1818 photoresist</li> <li>• 30 sec spin at 4,000 rpm</li> <li>• 1min @ 110°C hot plate bake</li> <li>• Turn off hot plate*</li> </ul>                                              |           |      |                            |                                            |              |
|              | <b>VIII. Expose 1818 – "Mask 1: Bottom Electrode"</b> <ul style="list-style-type: none"> <li>• Start EVG select 1818 process tab</li> <li>• Clean contact mask under solvent hood</li> <li>• Follow steps to center wafer/align to mask</li> <li>• Expose using EVG aligner (7 sec)</li> </ul>                                                                                                                                                           |           |      |                            |                                            |              |

Process Step #2

Revision 0 (29 Jan 2016)

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Figure A-1. Process follower for P(VDF-TrFE) membrane fabrication

| Wafer<br>SOI | Purpose<br>SOI - MEMBRANE                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | ID<br>SOI | Mask | Process<br>Membrane Device | Print Date<br>11/15/2016 |
|--------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------|------|----------------------------|--------------------------|
|              | <b>IX. Develop 1818</b> <ul style="list-style-type: none"> <li>Place wafer on base hood spinner</li> <li>45sec develop with 351:DI (1:5) at 500rpm (start-stop method)</li> <li>30 sec DI rinse at 500rpm</li> <li>Dry with nitrogen at 500rpm.</li> <li>Dry on tex wipe with nitrogen</li> <li>Verify develop under microscope, repeat this block if necessary</li> <li>Record thickness of the patterned 1818 on profilometer.</li> </ul>                                                                                                                                               |           |      |                            |                          |
|              | <b>X. Evaporate Ti/Al</b> <ul style="list-style-type: none"> <li>Deposit 250Å Ti, 3000Å Al, 250Å Au</li> </ul>                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |           |      |                            |                          |
|              | <b>XI. Release (lift-off bottom electrode metal)</b> <ul style="list-style-type: none"> <li>Using clear tape perform bulk removal of patterned metal</li> <li>10 min sample soak in acetone followed by 5min ultrasonic agitation</li> <li>30 sec acetone rinse at 500rpm</li> <li>30 sec methanol rinse at 500rpm</li> <li>Rinse sample and dry with nitrogen</li> </ul>                                                                                                                                                                                                                 |           |      |                            |                          |
|              | <b>XII. PVDF Deposition &amp; Anneal</b> <ul style="list-style-type: none"> <li>Turn on PECVD &amp; pumps and set chamber temperature to 110°C</li> <li>Place wafer (polished side up)</li> <li>Flood with 8% (PVDF-TrFE/MEK solution)</li> <li>30 sec spin at 2,000 rpm (for ~1µm thickness)</li> <li>Load sample in PECVD (~50mT) -100°C for 5 hours (DO NOT RUN PROFILE)</li> <li>Once 5hrs have elapsed, set PECVD chamber temperature to 27°C. Allow sample to cool down to room temperature (+/- 5°C)</li> <li>Unload sample from PECVD and power down PECVD &amp; pumps</li> </ul> |           |      |                            |                          |
|              | <b>XIII. Photoresist Application</b> <ul style="list-style-type: none"> <li>Photoresist(1818-electro-active polymer patterning):</li> <li>Turn on hot plate and set to 110°C*</li> <li>Place wafer on spinner (polished side up)</li> <li>Flood with 1818 photoresist</li> <li>30 sec spin at 4,000 rpm</li> <li>1min @ 110°C hot plate bake</li> <li>Turn off hot plate*</li> </ul>                                                                                                                                                                                                      |           |      |                            |                          |
|              | <b>XIV. Expose 1818 - "Mask 2: PVDF"</b> <ul style="list-style-type: none"> <li>Start EVG select 1818 process tab</li> <li>Clean contact mask under solvent hood</li> <li>Follow steps to center wafer/align to mask</li> <li>Expose using EVG aligner (7 sec)</li> </ul>                                                                                                                                                                                                                                                                                                                 |           |      |                            |                          |
|              | <b>XV. Develop 1818</b> <ul style="list-style-type: none"> <li>Place wafer on base hood spinner</li> <li>45sec develop with 351:DI (1:5) at 500rpm</li> <li>30 sec DI rinse at 500rpm</li> <li>Dry with nitrogen at 500rpm.</li> <li>Dry on tex wipe with nitrogen</li> <li>Verify develop under microscope, repeat this block if necessary</li> <li>Record thickness of the patterned 1818 using profilometer.</li> </ul>                                                                                                                                                                |           |      |                            |                          |

Figure A-2. Process follower for P(VDF-TrFE) membrane fabrication

| Wafer<br>SOI | Purpose<br>SOI - MEMBRANE                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | ID<br>SOI | Mask | Process<br>Membrane Device | Print Date<br>11/15/2016 |
|--------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------|------|----------------------------|--------------------------|
|              | <b>XVI. Reactive Ion Etch (RIE)</b> <ul style="list-style-type: none"> <li>From step XV, calculate etch time based on the recorded 1818 thickness (~123nm/min) - add 5 seconds to calculated time</li> <li>RIE etch for the calculated time at 75 sccm Oxygen gas environment with 100W RF power and 50 mT pressure (350VDC bias for directionality if using AFRL's processing equipment)</li> <li>Record RIE parameters: _____</li> <li>Record thickness of patterned PVDF using profilometer.</li> </ul>                                                                                                        |           |      |                            |                          |
|              | <b>XVII. Evaporate Cr/Au</b><br>Deposit 500Å Cr, 2000Å Au                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |           |      |                            |                          |
|              | <b>XVIII. Photoresist Application</b> <ul style="list-style-type: none"> <li>Photoresist(1818- top electrode patterning):</li> <li>Turn on hot plate and set to 110°C*</li> <li>Place wafer on spinner (polished side up)</li> <li>Flood with 1818 photoresist</li> <li>30 sec spin at 4,000 rpm</li> <li>1min @ 110°C hot plate bake</li> <li>Turn off hot plate*</li> </ul>                                                                                                                                                                                                                                     |           |      |                            |                          |
|              | <b>XIX. Expose 1818 - "Mask 3: Top Electrode"</b> <ul style="list-style-type: none"> <li>Start EVG select 1818 process tab</li> <li>Clean contact mask under solvent hood</li> <li>Follow steps to center wafer/align to mask</li> <li>Expose using EVG aligner (7 sec)</li> </ul>                                                                                                                                                                                                                                                                                                                                |           |      |                            |                          |
|              | <b>XX. Develop 1818</b> <ul style="list-style-type: none"> <li>Place wafer on base hood spinner</li> <li>45sec develop with 351:DI (1:5) at 500rpm</li> <li>30 sec DI rinse at 500rpm</li> <li>Dry with nitrogen at 500rpm.</li> <li>Dry on tex wipe with nitrogen</li> <li>Verify develop under microscope, repeat this block if necessary</li> <li>Record thickness of the patterned 1818 using profilometer.</li> </ul>                                                                                                                                                                                        |           |      |                            |                          |
|              | <b>XXI. Gold Etch (top electrode)</b> <ul style="list-style-type: none"> <li>Partially fill small container with Gold Etchant (enough to cover sample).</li> <li>Submerge sample in Gold Etch for 73 seconds. DO NOT AGITATE</li> <li>30 second rinse and dry with nitrogen.</li> <li>Inspect under microscope</li> <li>If not fully resolved - place in Gold Etch in 1 second intervals (be careful not to over-etch)</li> <li>Partially fill small container with CR-44 (enough to cover sample).</li> <li>Submerge sample in CR-44 for 10 seconds..</li> <li>30 second rinse and dry with nitrogen.</li> </ul> |           |      |                            |                          |
|              | <b>XXII. Photoresist Application (in preparation for DRIE)</b> <ul style="list-style-type: none"> <li>Turn on hot plate and set to 110°C*</li> <li>Place wafer on spinner (polished side up)</li> <li>Flood with 1818 photoresist</li> <li>30 sec spin at 4,000 rpm</li> <li>1min @ 110°C hot plate bake</li> <li>Turn off hot plate*</li> </ul>                                                                                                                                                                                                                                                                  |           |      |                            |                          |

Figure A-3. Process follower for P(VDF-TrFE) membrane fabrication

| Wafer<br>SOI | Purpose<br>SOI - MEMBRANE                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | ID<br>SOI | Mask | Process<br>Membrane Device | Print Date<br>11/15/2016 |
|--------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------|------|----------------------------|--------------------------|
|              | <b>XXIII. Apply SU8 (backside of sample)</b> <ul style="list-style-type: none"> <li>• Dropper SU8 over sample (do not let dropper touch edge of bottle)</li> <li>• Ensure sample is completely covered to the edges</li> <li>• 4 sec 500 rpm</li> <li>• 30 sec 2800 rpm gives 17microns approx (2000 rpm as backup)</li> <li>• Clean back of sample with acetone on a swab after sample finishes spinning</li> <li>• 3 min 65°C hot plate bake</li> <li>• 7 min 65°C hot plate bake</li> </ul> |           |      |                            |                          |
|              | <b>XXIV. Backside Align and Expose SU8 - "Mask 4: DRIE Layer"</b> <ul style="list-style-type: none"> <li>• Start EVG select SU8 process tab</li> <li>• Clean contact mask under solvent hood</li> <li>• Follow steps to center wafer/backside align to mask</li> <li>• Expose using EVG aligner (23 sec)</li> <li>• 1 min 65°C hot plate post exposure bake</li> <li>• 3 min 95°C hot plate post exposure bake</li> </ul>                                                                      |           |      |                            |                          |
|              | <b>XXV. Develop SU8</b> <ul style="list-style-type: none"> <li>• Partially fill small container with SU-8 developer. (enough to cover sample).</li> <li>• Submerge sample in developer for 4 minutes. Slight agitation</li> <li>• 30 sec DI rinse</li> <li>• Dry with nitrogen on tex wipe</li> <li>• Record thickness of the patterned SU8 using profilometer.</li> </ul>                                                                                                                     |           |      |                            |                          |
|              | <b>XXVI. DRIE</b> <ul style="list-style-type: none"> <li>• Place sample on carrier wafer</li> <li>• Backside DRIE to within 10-25um of Si Handle</li> </ul>                                                                                                                                                                                                                                                                                                                                    |           |      |                            |                          |
|              | <b>XVII. Post DRIE Processing</b> <ul style="list-style-type: none"> <li>• Repeat step XVI to remove protective 1818 photoresist</li> </ul>                                                                                                                                                                                                                                                                                                                                                    |           |      |                            |                          |
|              | <b>Inspect finished devices under optical microscope</b>                                                                                                                                                                                                                                                                                                                                                                                                                                       |           |      |                            |                          |

Figure A-4. Process follower for P(VDF-TrFE) membrane fabrication

## A-2 P(VDF-TrFE) Electroactive Polymer Process Follower

| Wafer<br>TEST | Purpose<br>EAP - MEMBRANE                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               | ID<br>EAP | Mask | Process<br>EAP Device                      | Print Date<br>11/15/2016 |
|---------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------|------|--------------------------------------------|--------------------------|
| Init.         | Process Step                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |           |      | Notes                                      | Date<br>Time             |
|               | <b>I. Solvent Clean (by hand)</b> <ul style="list-style-type: none"> <li>• Turn on hot plate and set to 110°C</li> <li>• 30 sec acetone rinse</li> <li>• 30 sec methanol rinse</li> <li>• 30 sec DI water rinse</li> <li>• Dry with nitrogen on texwipe</li> <li>• 2 min 110°C hot plate bake</li> <li>• Turn off hot plate*</li> </ul>                                                                                                                                                                                                                                                                 |           |      | <u>Start Date</u><br><br><u>Start Time</u> |                          |
|               | <b>II. Photoresist Application</b> <ul style="list-style-type: none"> <li>• Photoresist(1818- bottom electrode patterning &amp; lift-off):</li> <li>• Turn on hot plate and set to 110°C*</li> <li>• Place wafer on spinner (polished side up)</li> <li>• Flood with 1818 photoresist</li> <li>• 30 sec spin at 4,000 rpm</li> <li>• 1min @ 110°C hot plate bake</li> <li>• Turn off hot plate*</li> </ul>                                                                                                                                                                                              |           |      |                                            |                          |
|               | <b>III. Expose 1818 – "Mask 1: EAP Bottom Electrode"</b> <ul style="list-style-type: none"> <li>• Start EVG select 1818 process tab</li> <li>• Clean contact mask under solvent hood</li> <li>• Follow steps to center wafer/align to mask</li> <li>• Expose using EVG aligner (7 sec)</li> </ul>                                                                                                                                                                                                                                                                                                       |           |      |                                            |                          |
|               | <b>IV. Develop 1818</b> <ul style="list-style-type: none"> <li>• Place wafer on base hood spinner</li> <li>• 45sec develop with 351:DI (1:5) at 500rpm (start-stop method)</li> <li>• 30 sec DI rinse at 500rpm</li> <li>• Dry with nitrogen at 500rpm.</li> <li>• Dry on tex wipe with nitrogen</li> <li>• Verify develop under microscope, repeat this block if necessary</li> <li>• Record thickness of the patterned 1818 on profilometer.</li> </ul>                                                                                                                                               |           |      |                                            |                          |
|               | <b>V. Evaporate Ti/Al/Au</b> <ul style="list-style-type: none"> <li>• Deposit 500Å Ti, 3000Å Al, 250Å Au</li> </ul>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |           |      |                                            |                          |
|               | <b>VI. Release (lift-off bottom electrode metal)</b> <ul style="list-style-type: none"> <li>• Using clear tape perform bulk removal of patterned metal</li> <li>• 10 min sample soak in acetone followed by 5min ultrasonic agitation</li> <li>• 30 sec acetone rinse at 500rpm</li> <li>• 30 sec methanol rinse at 500rpm</li> <li>• Rinse sample and dry with nitrogen</li> </ul>                                                                                                                                                                                                                     |           |      |                                            |                          |
|               | <b>VII. PVDF Deposition &amp; Anneal</b> <ul style="list-style-type: none"> <li>• Turn on PECVD &amp; pumps and set chamber temperature to 110°C</li> <li>• Place wafer (polished side up)</li> <li>• Flood with 8% (PVDF-TrFE/MEK solution)</li> <li>• 30 sec spin at 2,000 rpm (for ~1µm thickness)</li> <li>• Load sample in PECVD (~50mT) -100°C for 5 hours (DO NOT RUN PROFILE)</li> <li>• Once 5hrs have elapsed, set PECVD chamber temperature to 27°C. Allow sample to cool down to room temperature (+/- 5°C)</li> <li>• Unload sample from PECVD and power down PECVD &amp; pumps</li> </ul> |           |      |                                            |                          |

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Figure A-5. Process follower for P(VDF-TrFE) EAP test coupon fabrication

| Wafer<br>TEST | Purpose<br>EAP - MEMBRANE                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | ID<br>EAP | Mask | Process<br>EAP Device | Print Date<br>11/15/2016 |
|---------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------|------|-----------------------|--------------------------|
|               | <b>VIII. Photoresist Application</b> <ul style="list-style-type: none"> <li>Photoresist(1818-electro-active polymer patterning):</li> <li>Turn on hot plate and set to 110°C*</li> <li>Place wafer on spinner (polished side up)</li> <li>Flood with 1818 photoresist</li> <li>30 sec spin at 4,000 rpm</li> <li>1min @ 110°C hot plate bake</li> <li>Turn off hot plate*</li> </ul>                                                                                                                      |           |      |                       |                          |
|               | <b>IX. Expose 1818 – "Mask 2: EAP PVDF"</b> <ul style="list-style-type: none"> <li>Start EVG select 1818 process tab</li> <li>Clean contact mask under solvent hood</li> <li>Follow steps to center wafer/align to mask</li> <li>Expose using EVG aligner (7 sec)</li> </ul>                                                                                                                                                                                                                              |           |      |                       |                          |
|               | <b>X. Develop 1818</b> <ul style="list-style-type: none"> <li>Place wafer on base hood spinner</li> <li>45sec develop with 351:DI (1:5) at 500rpm</li> <li>30 sec DI rinse at 500rpm</li> <li>Dry with nitrogen at 500rpm.</li> <li>Dry on tex wipe with nitrogen</li> <li>Verify develop under microscope, repeat this block if necessary</li> <li>Record thickness of the patterned 1818 using profilometer.</li> </ul>                                                                                 |           |      |                       |                          |
|               | <b>XI. Reactive Ion Etch (RIE)</b> <ul style="list-style-type: none"> <li>From step XV, calculate etch time based on the recorded 1818 thickness (~123nm/min) - add 5 seconds to calculated time</li> <li>RIE etch for the calculated time at 75 sccm Oxygen gas environment with 100W RF power and 50 mT pressure (350VDC bias for directionality if using AFRL's processing equipment)</li> <li>Record RIE parameters: _____</li> <li>Record thickness of patterned PVDF using profilometer.</li> </ul> |           |      |                       |                          |
|               | <b>XII. Evaporate Cr/Au</b> <ul style="list-style-type: none"> <li>Deposit 500Å Cr, 2000Å Au</li> </ul>                                                                                                                                                                                                                                                                                                                                                                                                   |           |      |                       |                          |
|               | <b>XIII. Photoresist Application</b> <ul style="list-style-type: none"> <li>Photoresist(1818- top electrode patterning):</li> <li>Turn on hot plate and set to 110°C*</li> <li>Place wafer on spinner (polished side up)</li> <li>Flood with 1818 photoresist</li> <li>30 sec spin at 4,000 rpm</li> <li>1min @ 110°C hot plate bake</li> <li>Turn off hot plate*</li> </ul>                                                                                                                              |           |      |                       |                          |
|               | <b>XIV. Expose 1818 – "Mask 3: EAP Top Electrode"</b> <ul style="list-style-type: none"> <li>Start EVG select 1818 process tab</li> <li>Clean contact mask under solvent hood</li> <li>Follow steps to center wafer/align to mask</li> <li>Expose using EVG aligner (7 sec)</li> </ul>                                                                                                                                                                                                                    |           |      |                       |                          |
|               | <b>XV. Develop 1818</b> <ul style="list-style-type: none"> <li>Place wafer on base hood spinner</li> <li>45sec develop with 351:DI (1:5) at 500rpm</li> <li>30 sec DI rinse at 500rpm</li> <li>Dry with nitrogen at 500rpm.</li> <li>Dry on tex wipe with nitrogen</li> <li>Verify develop under microscope, repeat this block if necessary</li> <li>Record thickness of the patterned 1818 using profilometer.</li> </ul>                                                                                |           |      |                       |                          |

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Figure A-6. Process follower for P(VDF-TrFE) EAP test coupon fabrication

## A-3 PolyMUMPs Post Fabrication Process Follower

| Wafer | Purpose                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | ID  | Mask  | Process                                    | Print Date |
|-------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|-------|--------------------------------------------|------------|
| MUMPs | PolyMUMPs Post Proc.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   | 117 | MUMPs | Post Processing                            | 1/10/2017  |
| Init. | Process Step                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |     |       | Notes                                      | Date Time  |
|       | <b>I. Solvent Clean (by hand)</b> <ul style="list-style-type: none"> <li>• Turn on hot plate and set to 110°C</li> <li>• 30 sec acetone rinse</li> <li>• 30 sec methanol rinse</li> <li>• 30 sec DI water rinse</li> <li>• Dry with nitrogen on texwipe</li> <li>• 2 min 110°C hot plate bake</li> <li>• Turn off hot plate*</li> </ul>                                                                                                                                                                                                                                                                                |     |       | <u>Start Date</u><br><br><u>Start Time</u> |            |
|       | <b>II. PVDF Deposition &amp; Anneal</b> <ul style="list-style-type: none"> <li>• Turn on PECVD &amp; pumps and set chamber temperature to 110°C</li> <li>• Place MUMPs sample on spinner (device side up)</li> <li>• Flood with 8% (PVDF-TrFE/MEK solution)</li> <li>• 30 sec spin at 2,000 rpm (for ~1µm thickness)</li> <li>• Load sample in PECVD (~50mT) ~100°C for 5 hours (DO NOT RUN PROFILE)</li> <li>• Once 5hrs have elapsed, set PECVD chamber temperature to 27°C. Allow sample to cool down to room temperature (+/- 5°C)</li> <li>• Unload sample from PECVD and power down PECVD &amp; pumps</li> </ul> |     |       |                                            |            |
|       | <b>III. Evaporate Cr/Au</b> <ul style="list-style-type: none"> <li>• Deposit 250Å Cr, 2000Å Au</li> </ul>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |     |       |                                            |            |
|       | <b>IV. Photoresist Application</b> <ul style="list-style-type: none"> <li>• Photoresist(1818- top electrode patterning):</li> <li>• Turn on hot plate and set to 110°C*</li> <li>• Place wafer on spinner (polished side up)</li> <li>• Flood with 1818 photoresist</li> <li>• 30 sec spin at 4,000 rpm</li> <li>• 1min @ 110°C hot plate bake</li> <li>• Turn off hot plate*</li> </ul>                                                                                                                                                                                                                               |     |       |                                            |            |
|       | <b>V. Expose 1818 – "Mask 0: MUMPs"</b> <ul style="list-style-type: none"> <li>• Start EVG select 1818 process tab</li> <li>• Clean contact mask under solvent hood</li> <li>• Follow steps to center wafer/align to mask</li> <li>• Expose using EVG aligner (7 sec)</li> </ul>                                                                                                                                                                                                                                                                                                                                       |     |       |                                            |            |
|       | <b>VI. Develop 1818</b> <ul style="list-style-type: none"> <li>• Place wafer on base hood spinner</li> <li>• 45sec develop with 351:DI (1:5) at 500rpm</li> <li>• 30 sec DI rinse at 500rpm</li> <li>• Dry with nitrogen at 500rpm.</li> <li>• Dry on tex wipe with nitrogen</li> <li>• Verify develop under microscope, repeat this block if necessary</li> <li>• Record thickness of the patterned 1818 using profilometer.</li> </ul>                                                                                                                                                                               |     |       |                                            |            |

Process Step #2

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Figure A-7. Process follower for PolyMUMPs P(VDF-TrFE) post fabrication

| Wafer | Purpose                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            | ID  | Mask  | Process         | Print Date |
|-------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|-------|-----------------|------------|
| MUMPs | PolyMUMPs Post Proc.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               | 117 | MUMPs | Post Processing | 1/10/2017  |
|       | VII. Metal Etch (top electrode)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |     |       |                 |            |
|       | <ul style="list-style-type: none"><li>Partially fill small container with Gold Etchant (enough to cover sample).</li><li>Submerge sample in Gold Etch for 73 seconds. DO NOT AGITATE</li><li>30 second rinse and dry with nitrogen.</li><li>Inspect under microscope</li><li>If not fully resolved – place in Gold Etch in 1 second intervals (be careful not to over-etch)</li><li>Partially fill small container with BOE. (enough to cover sample).</li><li>Submerge sample in CR-44 for 1-2 seconds..</li><li>30 second rinse and dry with nitrogen.</li><li>Record thickness of patterned Au/Cr using profilometer.</li></ul> |     |       |                 |            |
|       | VIII. Reactive Ion Etch (RIE)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |     |       |                 |            |
|       | <ul style="list-style-type: none"><li>From step VII, calculate etch time based on the recorded 1818 thickness (~123nm/min) - add 5 seconds to calculated time</li><li>RIE etch for the calculated time at 75 sccm Oxygen gas environment with 100W RF power and 50 mT pressure (350VDC bias for directionality if using AFRL's processing equipment)</li><li>Record RIE parameters: _____</li><li>Record thickness of patterned PVDF/Au/Ti using profilometer.</li></ul>                                                                                                                                                           |     |       |                 |            |
|       | IX. Device Release (Acid Hood)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |     |       |                 |            |
|       | <ul style="list-style-type: none"><li>Submerge sample in 49% Hydrofluoric acid (49% HF)</li><li>Soak for 15 minutes</li><li>Submerge sample in DI water for 2-3 minutes</li><li>Submerge sample in 3-stage Isopropyl Alcohol dip (1-2 min each stage)</li><li>Place sample in transport beaker, submerged in Isopropyl Alcohol</li></ul>                                                                                                                                                                                                                                                                                           |     |       |                 |            |
|       | X. CO2 Dry (Breezeway)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |     |       |                 |            |
|       | <ul style="list-style-type: none"><li>Place sample in drying chamber</li><li>Fill chamber with enough Isopropyl Alcohol to cover sample</li><li>Close chamber and run dry cycle</li></ul>                                                                                                                                                                                                                                                                                                                                                                                                                                          |     |       |                 |            |
|       | Inspect finished devices under optical microscope                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |     |       |                 |            |

Figure A-8. Process follower for PolyMUMPs P(VDF-TrFE) post fabrication

## Appendix B.

### B-1 SOI Membrane Masks

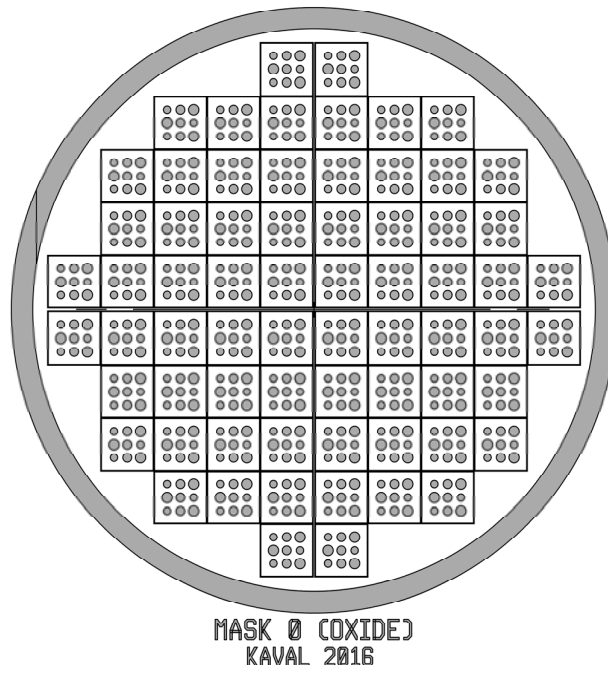


Figure B-1. Mask Layer 0 - Patterning for oxide etch

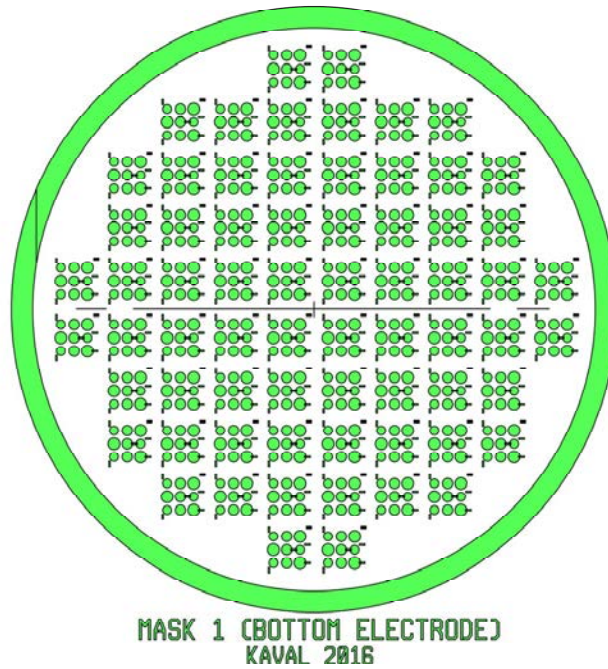


Figure B-2. Mask Layer 1 - Patterning for bottom electrode lift-off

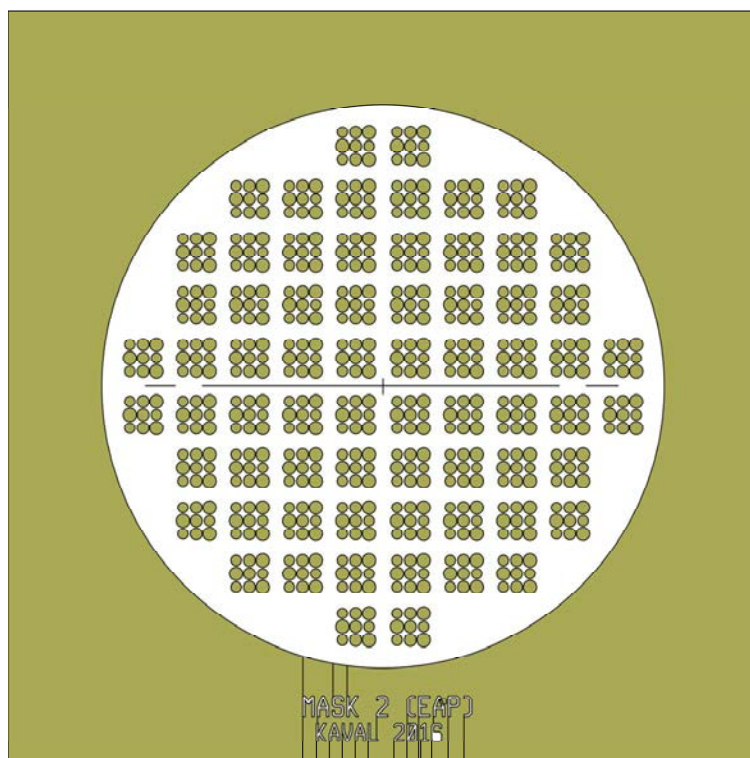


Figure B-3. Mask Layer 2 - Patterning for P(VDF-TrFE) RIE



Figure B-4. Mask Layer 3 - Patterning for top electrode wet etch

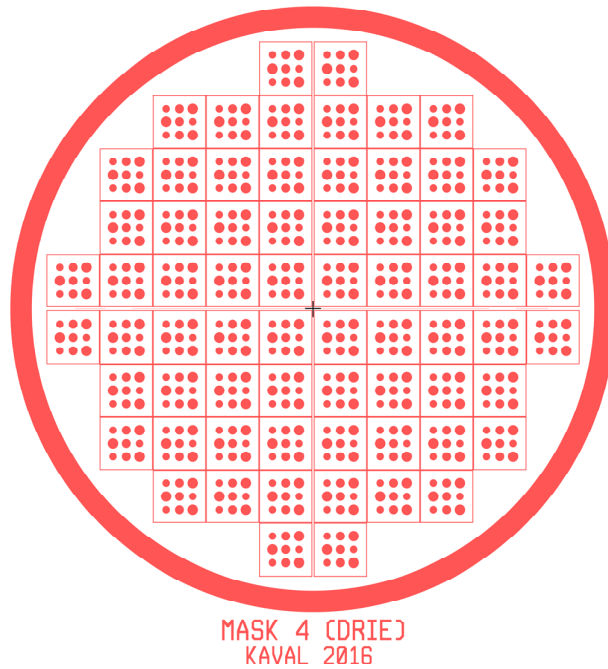


Figure B-5. Mask Layer 4 - Patterning for backside DRIE

## B-2 Electroactive Polymer Characterization Masks

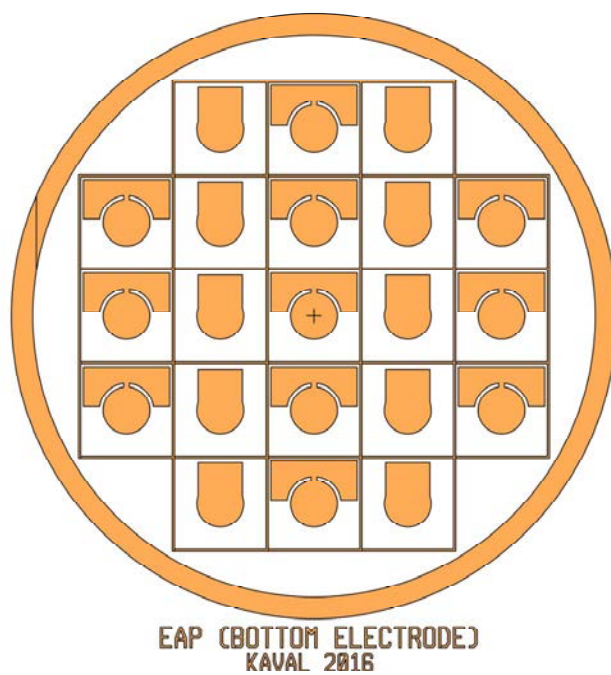


Figure B-6. Mask Layer 1 - Patterning for bottom electrode lift-off



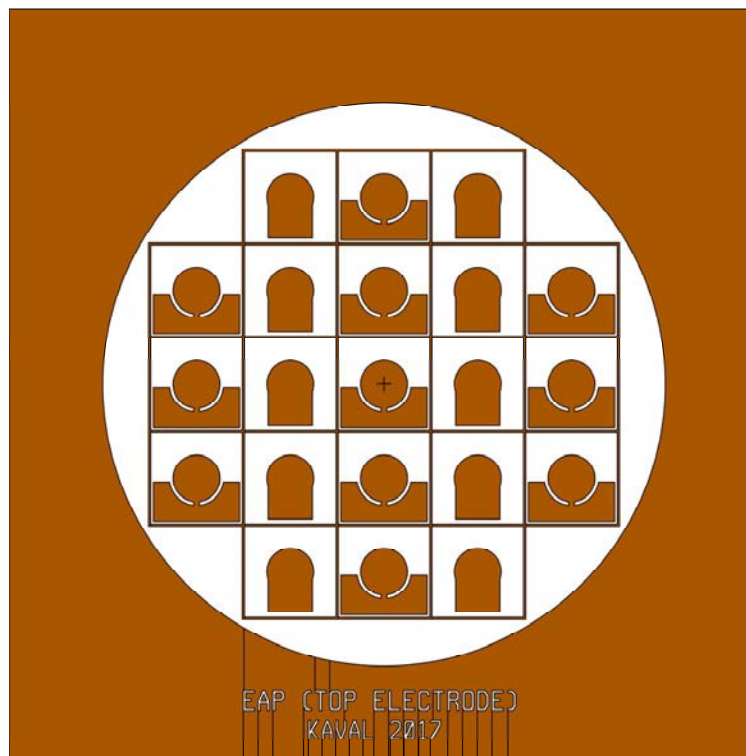


Figure B-8. Mask Layer 3 - Patterning for top electrode wet etch

### B-3 PolyMUMPs Post-processing Mask (pre-release)

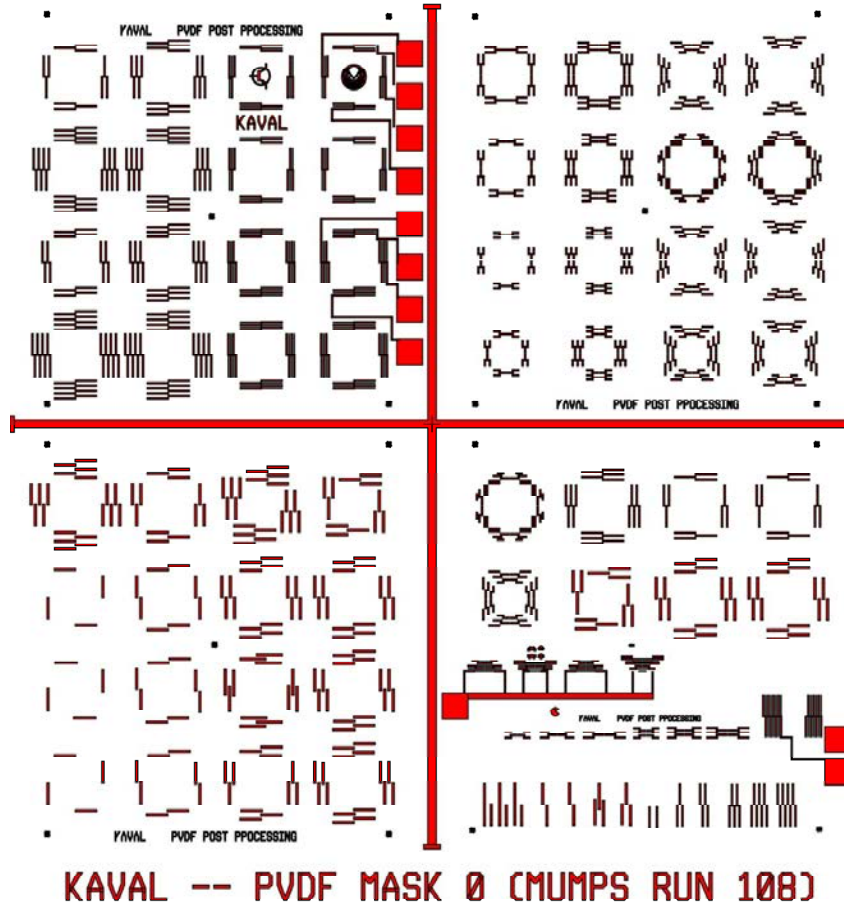


Figure B-9. Patterning for PolyMUMPs top electrode and P(VDF-TrFE) - (wet metal etch followed by RIE)

## Appendix C.

### C-1 Heidelberg $\mu$ PG 101 Mask Writer



Figure C-1. Heidelberg  $\mu$ PG 101 Mask Writer used for printing 4" masks used in this research

## C-2 Alpha-Step IQ



Figure C-2. Alpha-Step IQ surface profilometer

### C-3 EVG®620 Automated Mask Alignment System



Figure C-3. The EVG®620 Automated Mask Alignment System is a tool designed for optical double-side lithography and precision alignment up to 150 mm wafer sizes.

C-4 Minilock-Phantom reactive ion etcher (RIE)/inductively coupled plasma (ICP)



**Figure C-4.** The Minilock-Phantom RIE ICP is an advanced high density etching system with a vacuum loadlock and an inductively coupled plasma (ICP) source used for etching polymers and dielectric materials. The ICP allows for improved anisotropy in high aspect ratio structures

## C-5 Unaxis 790 reactive ion etcher (RIE)



**Figure C-5.** The Unaxis 790 reactive ion etcher (RIE) is a general-purpose parallel plate plasma etching system which uses ionized fluorocarbon gases and oxygen to etch polymer and dielectric materials. It can also use argon to physically etch metals. The large chamber can accommodate substrates ranging from 200 mm diameter wafers down to small pieces

## C-6 Minilock-Orion III PECVD system



**Figure C-6.** Minilock-Orion is a plasma-enhanced chemical vapor deposition system. The heated vacuum loadlock stage of this system was utilized in order to anneal the P(VDF-TrFE) copolymer

## C-7 Quanta 3D 200i Scanning Electron Microscope

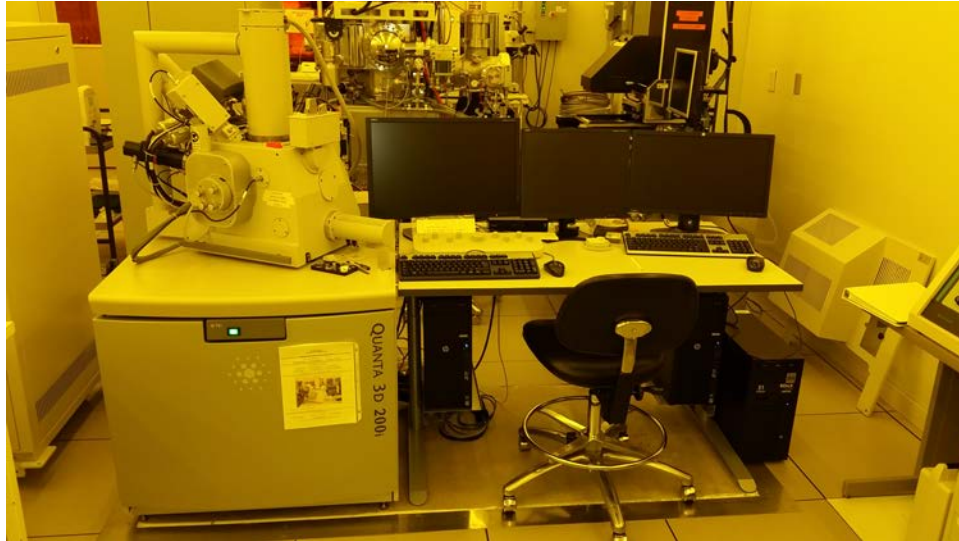


**Figure C-7.** The Quanta 3D 200i is a characterization instrument designed to examine materials and features operation in three modes: high-vacuum, low-vacuum (10-130 Pa) and environmental (130-2600 Pa) with a range of magnification from 10x to 100,000x. The microscope facilitates testing of conductive materials (metals and alloys) as well as non-conductive samples such as: ceramics, polymers and organic materials

## C-8 Blue M Standard Mechanical Convection Oven



Figure C-8. Blue M standard convection oven for curing, moisture drying, and other processes and testing requirements

## Appendix D.

### D-1 Sawyer Tower PCB

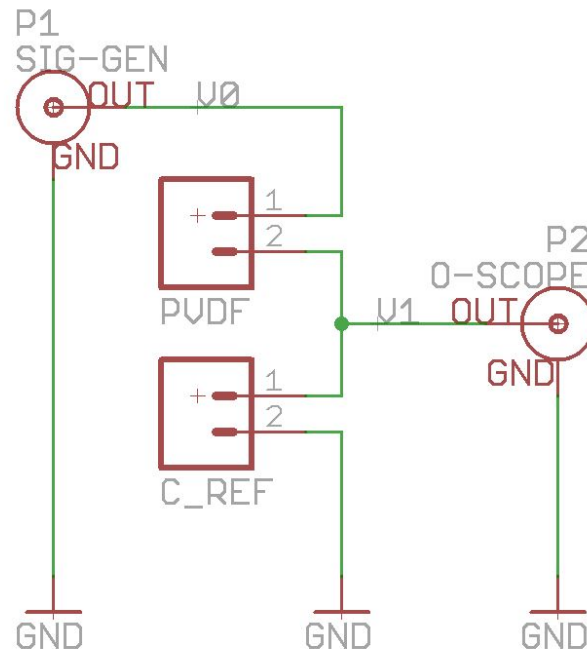


Figure D-1. Sawyer Tower Circuit Schematic

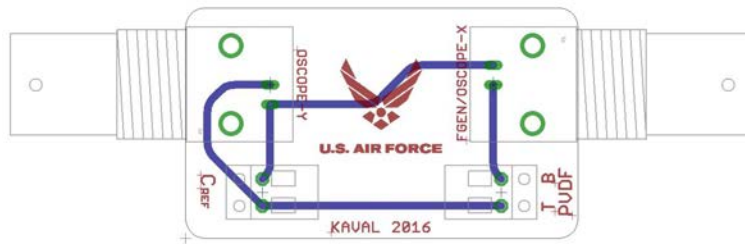


Figure D-2. Sawyer Tower PCB Layout

## D-2 Instrumentation Amplifier Circuit

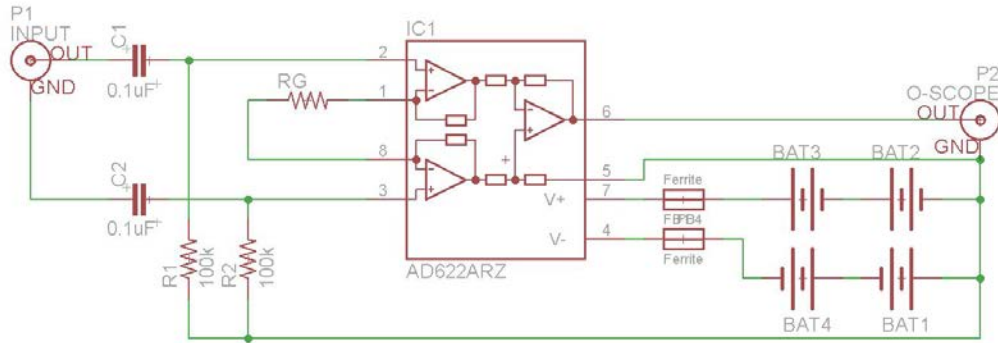
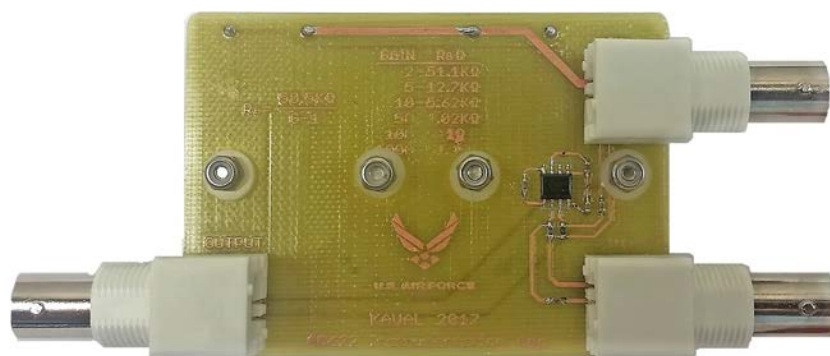


Figure D-3. Custom low-cost instrumentation amplifier to measure P(VDF-TrFE) with an input impedance of  $15\text{G}\Omega$

| Desired Gain | 1% Std Table Value of $R_G$ , $\Omega$ | Calculated Gain |
|--------------|----------------------------------------|-----------------|
| 2            | 51.1 k                                 | 1.988           |
| 5            | 12.7 k                                 | 4.976           |
| 10           | 5.62 k                                 | 9.986           |
| 20           | 2.67 k                                 | 19.91           |
| 33           | 1.58 k                                 | 32.96           |
| 40           | 1.3 k                                  | 39.85           |
| 50           | 1.02 k                                 | 50.50           |
| 65           | 787                                    | 65.17           |
| 100          | 511                                    | 99.83           |
| 200          | 255                                    | 199.0           |
| 500          | 102                                    | 496.1           |
| 1000         | 51.1                                   | 989.3           |

Figure D-4. Required values of  $R_G$  for various gains. Note that for  $G = 1$ , the  $R_G$  pins are unconnected ( $R_G = \infty$ ). For any arbitrary gain,  $R_G$  can be calculated by using the formula:  $R_G = \frac{50.5k\Omega}{G-1}$



(a) Front side



(b) Back side

**Figure D-5.** Custom fabricated instrumentation amplifier for piezoelectric evaluation by measuring the open loop voltage – Note: the preamplifier runs on batteries in order to eliminate external noise

### D-3 Piezoelectric Membrane Characterization Configuration

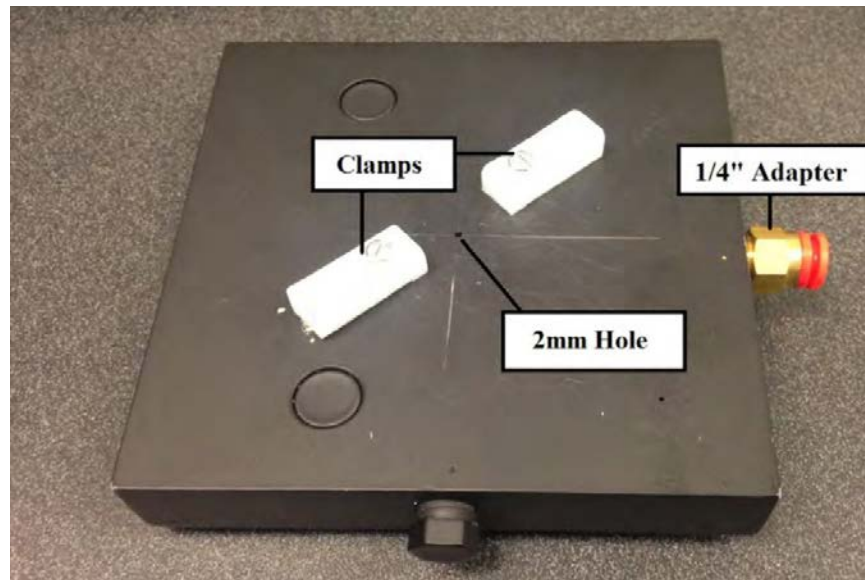


Figure D-6. Platform for holding samples in place while applying pressurized N<sub>2</sub> to the back of the pressure sensor during deflection measurement with the ZYGO.

[13]

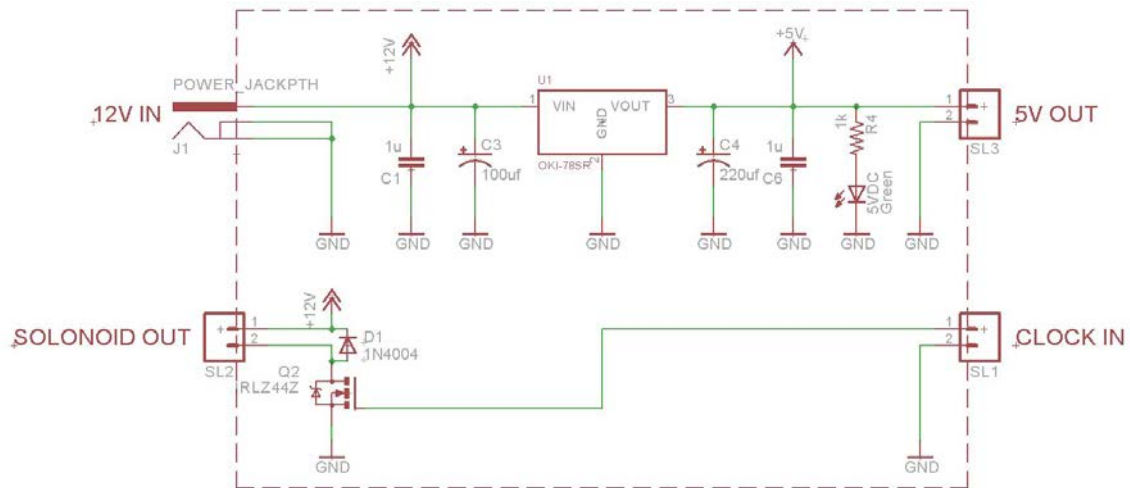


Figure D-7. Solenoid control valve circuit schematic - NI Analog Discovery 2 DAQ interface

#### D-4 Nanopower Energy Harvesting Power Supply

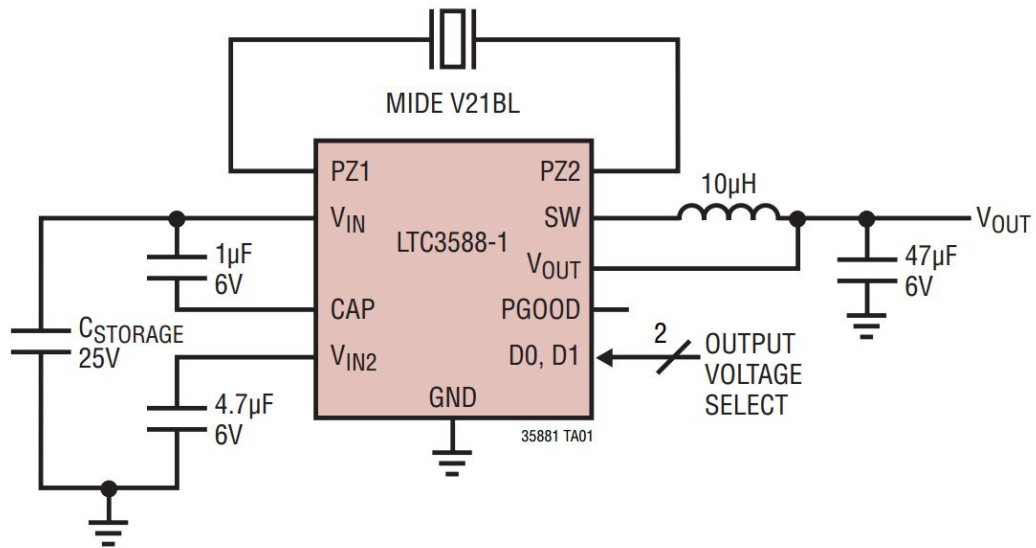


Figure D-8. Nanopower Energy Harvesting Power Supply

[44]

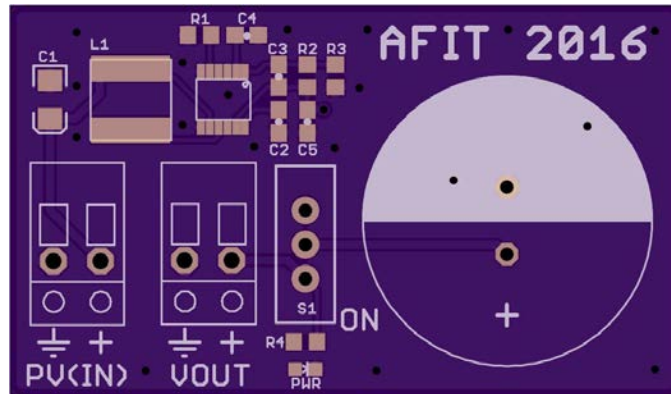


Figure D-9. Custom piezoelectric energy harvesting PCB

## D-5 Tinned copper foil tape used for electrode contact wiring

### 3M™ Tin-Plated Copper Foil with Conductive Adhesive Tape 1183

#### Data Sheet

January 2014

|                                                   |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |
|---------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <b>Description</b>                                | 3M™ Tin-Plated Copper Foil Tape 1183 consists of a 1-ounce dead soft tin-plated copper foil backing and an electrically conductive, pressure-sensitive acrylic adhesive. This tape is supplied on a removable liner for easy handling and die-cutting. It has excellent solderability.                                                                                                                                                                                                                                                                                                                 |
| <b>Agency Approvals &amp; Self Certifications</b> | Meets flame retardant requirements of UL510, Product Category OANZ2, and 3M File No. 17385<br><br>RoHS<br>2011/65/EC<br><br>"RoHS 2011/65/EU" means that the product or part does not contain any of the substances in excess of the maximum concentration values ("MCVs") in EU RoHS Directive 2011/65/EU. The MCVs are by weight in homogeneous materials. This information represents 3M's knowledge and belief, which may be based in whole or in part on information provided by third party suppliers to 3M.                                                                                     |
| <b>Applications</b>                               | Tin-Plated Copper Foil Tape 1183 is typically used for applications requiring excellent electrical conductivity from the application substrate through the adhesive to the foil backing. Common uses include grounding and EMI shielding in equipment, components, shielded rooms, etc. The tin plating facilitates soldering to the backing and resists oxidation and corrosion.                                                                                                                                                                                                                      |
| <b>Shielding Effectiveness</b>                    | Many factors determine the true shielding effectiveness of a shielding tape, including type and thickness of foil, adhesive type, intimacy of contact, smoothness of application surface, strength and frequency of the EMI signal, etc. However, using standard tests and fixtures, it is possible to determine a value for the attenuation. For 3M 1183 tape, typical shielding effectiveness (far field) is in the range of 70dB to 85dB (30 MHz to 1 GHz).                                                                                                                                         |
| <b>Specifications</b>                             | Tin-Plated Copper Foil Tape 1183 is a tin-plated copper foil tape with acrylic conductive pressure-sensitive adhesive.<br><br>The tape shall be coated on one side with pressure-sensitive adhesive, which shall not require heat, moisture or other preparation prior to or subsequent to application. The adhesive coating shall be smooth and uniform and be free of lumps and bare spots. There shall be no separator between adjacent layers of the roll. The tape shall perform at a temperature of -40F° (-40°C) to 266°F (130°C) without substantial loss of tensile or electrical properties. |

Figure D-10. 3M 1183 electronic specialty tape datasheet, pg 1 of 2

## 3M™ Tin-Plated Copper Foil with Conductive Adhesive Tape 1183

| Typical Properties                          | Not for specifications. Values are typical, not to be considered minimum or maximum. Properties measured at room temperature 73°F (-23°C) unless otherwise stated.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |                        |                                    |       |                                              |          |                    |                 |                   |                   |                  |                              |                    |                                    |         |                               |      |                                |                      |                                |                      |                   |                 |                                      |                                |
|---------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------|------------------------------------|-------|----------------------------------------------|----------|--------------------|-----------------|-------------------|-------------------|------------------|------------------------------|--------------------|------------------------------------|---------|-------------------------------|------|--------------------------------|----------------------|--------------------------------|----------------------|-------------------|-----------------|--------------------------------------|--------------------------------|
|                                             | <table> <tr> <th>Property (Test Method)</th><th>Typical Value<br/>US units (metric)</th></tr> <tr> <td>Color</td><td>Tin (foil may have a slight color variation)</td></tr> <tr> <td>Adhesive</td><td>Acrylic Conductive</td></tr> <tr> <td>Type of Backing</td><td>Tin-Plated Copper</td></tr> <tr> <td>Backing Thickness</td><td>1.4 mil (0.04mm)</td></tr> <tr> <td>Total Thickness (ASTM D1000)</td><td>2.6 mils (0.066mm)</td></tr> <tr> <td>Electrical Resistance<sup>1</sup></td><td>0.005 Ω</td></tr> <tr> <td>Flame Retardancy<sup>2</sup></td><td>Pass</td></tr> <tr> <td>Breaking Strength (ASTM D3759)</td><td>25 lbs/in (44N/10mm)</td></tr> <tr> <td>Adhesion Strength (ASTM D3330)</td><td>35 oz/in (3.8N/10mm)</td></tr> <tr> <td>Temperature Range</td><td>-40°C to +130°C</td></tr> <tr> <td>Shielding Effectiveness ( ASTM 4935)</td><td>70dB to 85 dB, 30 MHz to 1 GHz</td></tr> </table> <div> <div>1. MIL-STD-202 Method 303 maintained at 5 psi (3.4N/cm<sup>2</sup>) measured over 1 in<sup>2</sup> surface area. Conductive particles in the adhesive provide the electrical path between the application substrate and the foil backing.</div> <div>2. UL Recognized for flame retardancy per UL 510, Product Category OANZ2, File E17385</div> </div> | Property (Test Method) | Typical Value<br>US units (metric) | Color | Tin (foil may have a slight color variation) | Adhesive | Acrylic Conductive | Type of Backing | Tin-Plated Copper | Backing Thickness | 1.4 mil (0.04mm) | Total Thickness (ASTM D1000) | 2.6 mils (0.066mm) | Electrical Resistance <sup>1</sup> | 0.005 Ω | Flame Retardancy <sup>2</sup> | Pass | Breaking Strength (ASTM D3759) | 25 lbs/in (44N/10mm) | Adhesion Strength (ASTM D3330) | 35 oz/in (3.8N/10mm) | Temperature Range | -40°C to +130°C | Shielding Effectiveness ( ASTM 4935) | 70dB to 85 dB, 30 MHz to 1 GHz |
| Property (Test Method)                      | Typical Value<br>US units (metric)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |                        |                                    |       |                                              |          |                    |                 |                   |                   |                  |                              |                    |                                    |         |                               |      |                                |                      |                                |                      |                   |                 |                                      |                                |
| Color                                       | Tin (foil may have a slight color variation)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |                        |                                    |       |                                              |          |                    |                 |                   |                   |                  |                              |                    |                                    |         |                               |      |                                |                      |                                |                      |                   |                 |                                      |                                |
| Adhesive                                    | Acrylic Conductive                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |                        |                                    |       |                                              |          |                    |                 |                   |                   |                  |                              |                    |                                    |         |                               |      |                                |                      |                                |                      |                   |                 |                                      |                                |
| Type of Backing                             | Tin-Plated Copper                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                |                        |                                    |       |                                              |          |                    |                 |                   |                   |                  |                              |                    |                                    |         |                               |      |                                |                      |                                |                      |                   |                 |                                      |                                |
| Backing Thickness                           | 1.4 mil (0.04mm)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 |                        |                                    |       |                                              |          |                    |                 |                   |                   |                  |                              |                    |                                    |         |                               |      |                                |                      |                                |                      |                   |                 |                                      |                                |
| Total Thickness (ASTM D1000)                | 2.6 mils (0.066mm)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |                        |                                    |       |                                              |          |                    |                 |                   |                   |                  |                              |                    |                                    |         |                               |      |                                |                      |                                |                      |                   |                 |                                      |                                |
| Electrical Resistance <sup>1</sup>          | 0.005 Ω                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |                        |                                    |       |                                              |          |                    |                 |                   |                   |                  |                              |                    |                                    |         |                               |      |                                |                      |                                |                      |                   |                 |                                      |                                |
| Flame Retardancy <sup>2</sup>               | Pass                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |                        |                                    |       |                                              |          |                    |                 |                   |                   |                  |                              |                    |                                    |         |                               |      |                                |                      |                                |                      |                   |                 |                                      |                                |
| Breaking Strength (ASTM D3759)              | 25 lbs/in (44N/10mm)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |                        |                                    |       |                                              |          |                    |                 |                   |                   |                  |                              |                    |                                    |         |                               |      |                                |                      |                                |                      |                   |                 |                                      |                                |
| Adhesion Strength (ASTM D3330)              | 35 oz/in (3.8N/10mm)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |                        |                                    |       |                                              |          |                    |                 |                   |                   |                  |                              |                    |                                    |         |                               |      |                                |                      |                                |                      |                   |                 |                                      |                                |
| Temperature Range                           | -40°C to +130°C                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                        |                                    |       |                                              |          |                    |                 |                   |                   |                  |                              |                    |                                    |         |                               |      |                                |                      |                                |                      |                   |                 |                                      |                                |
| Shielding Effectiveness ( ASTM 4935)        | 70dB to 85 dB, 30 MHz to 1 GHz                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                        |                                    |       |                                              |          |                    |                 |                   |                   |                  |                              |                    |                                    |         |                               |      |                                |                      |                                |                      |                   |                 |                                      |                                |
| Shelf Life & Storage                        | This product has a 5-year shelf life from date of manufacture when stored in a humidity controlled storage (10°C/50°F to 27°C/80°F and <75% relative humidity).                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                        |                                    |       |                                              |          |                    |                 |                   |                   |                  |                              |                    |                                    |         |                               |      |                                |                      |                                |                      |                   |                 |                                      |                                |
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Figure D-11. 3M 1183 electronic specialty tape datasheet, pg 2 of 2

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## Vita

Captain William G. Kaval was born in Washington, D.C. and raised in Southern Maryland. In 1997 he enlisted in the Air Force as an Avionics Technician. He went on to earn his associate degree in avionics technology from the Community College of the Air Force in 2000. In 2005 he was selected for the Airman Education and Commissioning Program and earned his undergraduate degree in electrical engineering from the University of Maryland, College Park in 2008. While at UMD, he was a part-time staff member of the university's Air Force ROTC detachment. He received his commission as a second lieutenant in the Air Force in 2008 through the Officer Training School, Maxwell AFB, AL. William's first assignment upon commissioning was at the Air Force Research Laboratory at Wright Patterson Air Force Base in Dayton, Ohio where he researched smart structures and embedded electronics. His following assignment was to the 28th Test and Evaluation Squadron at Davis Monthan AFB in Tucson, Arizona. While in Tucson he worked as lead operational flight test engineer. In 2015, Captain Kaval returned to Wright Patterson AFB to the Air Force Institute of Technology as an MS student. He is a student section leader and embedded systems designer.

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| <b>14. ABSTRACT</b><br><br>Research of electrostrictive polymers has generated new opportunities for harvesting energy from the surrounding environment and converting it into usable electrical energy. Piezoelectric ceramic-based devices have long been used in energy harvesting for converting mechanical motion to electrical energy. Nevertheless, those materials tend to be unsuitable for low-frequency mechanical excitations such as human movement. Since organic polymers are typically softer and more flexible, the translated electrical energy output is considerably higher under the same mechanical force. Currently, investigations in using electroactive polymers for energy harvesting, and mechanical-to-electrical energy conversion, are beginning to show potential for this application. In this paper we discuss methods of energy harvesting using membrane structures and various methods used to convert it into usable energy. Since polymers are typically used in capacitive energy-harvesting designs, the uses of polymer materials with large relative permittivities have demonstrated success for mechanical to electrical energy conversion. Further investigations will be used to identify suitable micro-electro-mechanical systems (MEMs) structures given specific types of low-frequency mechanical excitations (10-100Hz). |                    |                     |                                          |                            |                                                                                               |  |
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| <b>16. SECURITY CLASSIFICATION OF:</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                    |                     | <b>17. LIMITATION OF ABSTRACT</b>        | <b>18. NUMBER OF PAGES</b> | <b>19a. NAME OF RESPONSIBLE PERSON</b>                                                        |  |
| <b>a. REPORT</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              | <b>b. ABSTRACT</b> | <b>c. THIS PAGE</b> |                                          |                            | Capt. Robert A. Lake, Ph.D.(ENG)                                                              |  |
| U                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | U                  | U                   | UU                                       | 152                        | <b>19b. TELEPHONE NUMBER (include area code)</b><br>(937) 255-3636 x4550 robert.lake@afit.edu |  |