

**ARTIFICIAL MUSCLE PROJECT:
PROCESS DEVELOPMENT OF POLYDIMETHYL
SILOXANE THIN FILMS FOR USE IN
DIELECTRIC ELECTROACTIVE POLYMER
ARTIFICIAL MUSCLE ACTUATORS**

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Abstract

An artificial muscle design was created founded on the principles of a dielectric electroactive polymer (DEAP), which is fundamentally similar to a capacitor. A polydimethyl siloxane (PDMS)-based elastomer, Sylgard 184 from Dow Corning, was chosen for the design and spun coat onto polystyrene (PS) Petri dishes at varying speeds to create a thin film, using speeds of 2000 rpm, 3000 rpm, 4000 rpm, 5000 rpm, and 6000 rpm. The film thicknesses were measured optically through use of a microscope with coupled computer imaging software to generate a characteristic curve of film thickness to spin speed, achieving a minimum film thickness of 15.9 microns at a spin speed of 6000 rpm. A second elastomer, 3-6121 Low Temperature Elastomer from Dow Corning, was attained and compounded at varying volume ratios with the primary Sylgard 184 in effort to obtain a polymer compound with a decreased compressive modulus, effectively reducing the required actuation voltage of the design. Varying polymer compounds were tested in compression and the compound of 80 vol% primary elastomer and 20 vol% secondary elastomer was found to have a drastically reduced modulus of compression. Thin films of pure primary elastomer and the reduced-modulus polymer compound were created for electrostatic actuation testing, measured via interferometry. Dielectric breakdown was observed during electrostatic testing before significant actuation was achieved. Mechanical actuation testing using .5 kg, 1 kg, 1.5 kg, and 2 kg masses was adopted to prove the concept. Mechanical testing proved successful, achieving a maximum actuation of 20% strain and 35% strain for the pure primary elastomer and the reduced-modulus polymer compound, respectively.

Keywords: Artificial muscle actuator, elastomer, polydimethyl siloxane, PDMS, dielectric electroactive polymer, DEAP, spin coating

Problem statement

High voltages are currently required to achieve significant actuation of DEAP actuators. This project focuses to reduce the required voltage for significant actuation through DEAP synthesis and processing techniques.

1. Introduction

1.1 Justification and Broader Impacts

Mechanical actuators, such as screw jacks and cams, provide purely manual operation that is cheap and repeatable. Adding a motor automates the operation. However, mechanical actuators have many moving parts and are prone to wear. Hydraulic motors can provide large movement and large forces, but are prone to leaks and typically require a separate hydraulic pump. Alternately, artificial muscle actuators have no moving parts, providing reliable and repeatable actuation without the moving parts or mechanisms, effectively limiting wear.

Aside from replacing actuating motors, artificial muscle actuators are under research for use in consumer electronics, space exploration, pumps, and medicine, among others.

Such as Artificial Muscle, Inc. who uses the technology to improve the ‘feel’ of touch-screens and imitate the sensation of typing on a keyboard with a touch-screen in a cellular

phone¹. Artificial muscles are also being tested as wiper blades to clear dust off of camera lenses on

planet exploration rovers, create gripping devices² (Figure 1), as well as actuate Braille dots on a

screen display³. The technology can create

diaphragm actuators, which can increase or decrease

pressure in a pump. In the field of medicine,

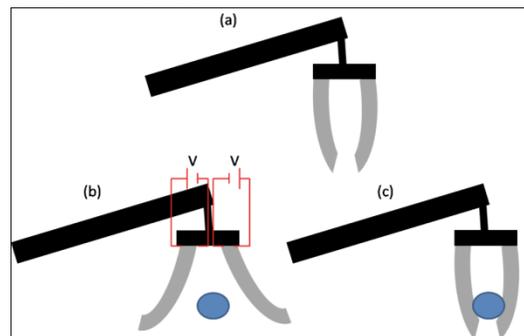


Figure 1. Cartoon drawing of an EAP gripping device. (a) A voltage is applied and the EAP fingers deform in order to surround the ball. (b) When the voltage is removed the EAP fingers return to their original shape and grip the ball. (c)

artificial muscle technology is being researched to aid persons diagnosed with muscular dystrophy and to regulate the distribution of drugs in an *in vivo* drug implant⁴.

1.2 Background

Historically, ceramic piezoelectrics have been the only viable materials for use as an artificial muscle actuator, providing small actuations in the fractions of a percent strain. However, artificial muscle actuator research has recently transitioned to focus on a special class of polymers called electroactive polymers (EAP), which undergo a change in either size or shape when stimulated by an electric field. In the late 1990s, a demonstration exhibited an EAP straining to 380% strain⁵, far more strain than ceramic piezoelectrics were ever capable of.

In 1999, Dr. Yoseph Bar-Cohen issued a scientific challenge to the worldwide research and engineering community to develop a robotic arm actuated by artificial muscles to win an arm-wrestling match with a human⁶. Since then, EAP research has blossomed, recognizing multiple EAP types: ferroelectric polymers, such as liquid crystalline polymers used in displays that crystallize in response to an electric field; ionic electroactive polymers whose is actuation caused by displacement of ions within the material; and dielectric electroactive polymers (DEAP).

1.3 Artificial Muscle Design

This artificial muscle project adopted the dielectric electroactive polymer (DEAP) as its design. DEAP actuation is the result of electrostatic forces between two electrodes compressing an insulating polymer⁷ (Figure 2), fundamentally similar to a capacitor.

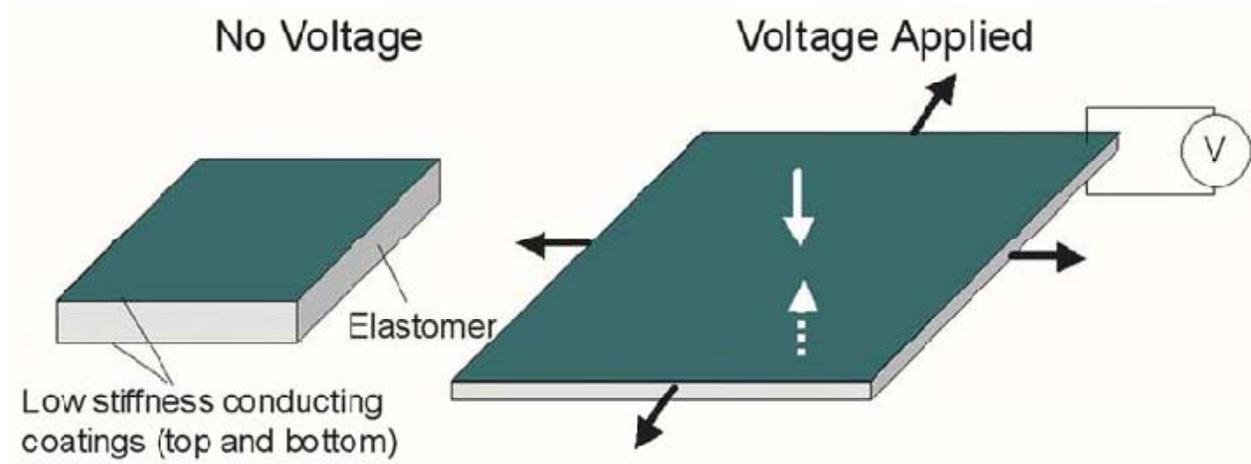


Figure 2. DEAP Actuator diagram showing change in dimensions⁷

This style of actuator generally requires a large actuation voltage, typically in the hundreds to thousands of volts, but has very low power consumption. The magnitude of this actuation stress produced by the DEAP is modeled by the Maxwell Tensor Equation⁷ (Eq. 1).

Eq. (1)

Maxwell Tensor Equation

$$P = \epsilon_0 \epsilon_r \left(\frac{V}{d}\right)^2$$

Where:

P = Stress (Pa)

ϵ_0 = Permittivity of Free Space

ϵ_r = Dielectric Constant

V = Voltage (Volts)

d = Film Thickness (m)

The amount of stress applied to the elastomer is affected primarily by the voltage that is applied and the film thickness. It is because of this that research has focused on creating

actuators with high voltage ranging from 1 kV-4 kV and low thicknesses of 100 microns⁷. Lowering the film thickness will allow the amount of voltage used to decrease.

An artificial muscle actuator will be comprised of stacks of individual actuation cells⁷ (Figure 3). The stack will alternate between a layer of electrode and a layer of DEAP material. To achieve biomimicry of the range of average biological muscle⁸ the DEAP stack has a goal to achieve a strain between 20% and 40%.

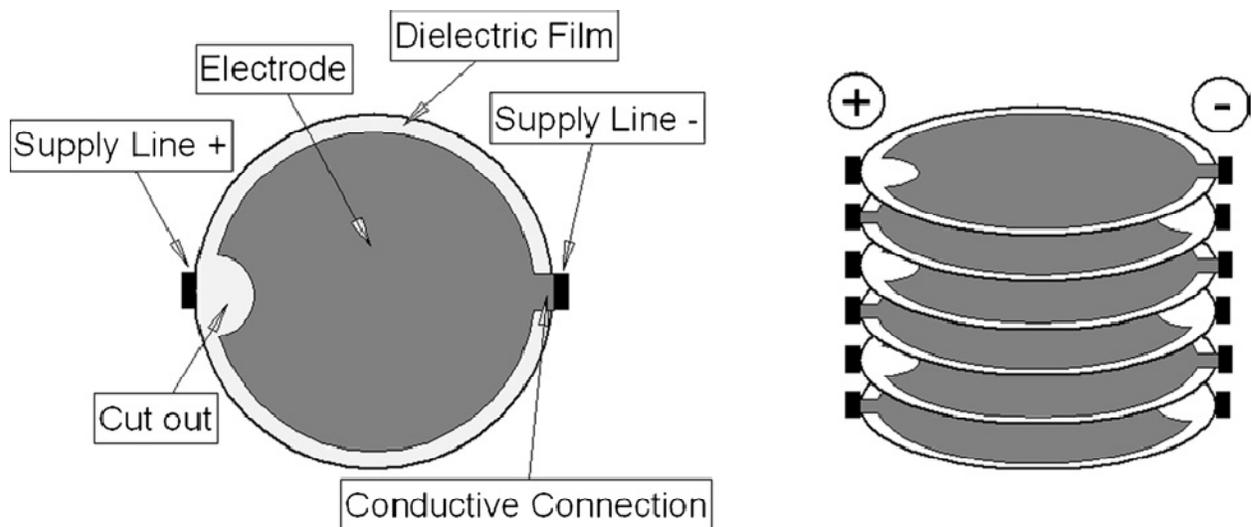


Figure 3. DEAP stack diagram⁷.

2. Materials

2.1 Material Constraints

This project required a clear, dielectric material with an elastic deformation region exceeding 20 % strain, a low modulus of compression, and a high dielectric breakdown strength. After selecting a primary material that met these criteria, a second material would be selected and used as an additive in the primary material to additionally lower its

modulus of compression. Ordinary actuation for a biological muscle is anywhere from 20 to 40 % strain, therefore the material must be capable of elastically deforming under at least 20 % strain. The actuation of the design depends on the compressive modulus of the material and its insulating an electric field, resulting in the criteria of a low compressive modulus and high dielectric breakdown strength respectively. Additionally, the material must have good light-throughput characteristics, i.e. clear, to allow for interferometry testing. Of the material options (metals, polymers, ceramics, and composites), polymers were a natural choice for their low modulus and insulative properties.

2.2 Polymer Background

A polymer is a macromolecule consisting of repeating structural units generally bonded covalently. The simplest form of a polymer is a linear chain without functional groups or branching, such as polyethylene. Functional groups are atoms within the polymer molecule that are covalently linked to the backbone of the polymer chain, but are not part of the backbone. They often are responsible for the characteristic chemical reactivity of the polymer. There are two noteworthy architectural features in polymers that affect the properties of the polymer: branching and crosslinking. Branches are substituent side chains, or branches, that are covalently linked to the backbone only. These branches can either be identical to the backbone or a different chain, as in the case of a grafted copolymer. Some common branched polymer architectures include star, comb, brush, graft, and dendrimer polymers.



Figure 4. Structural schematic of a crosslinked polymer.

Unlike branching, crosslinking is the formation of covalent bonds between two polymer

chains (Figure 4). Crosslinking generally increases the glass transition temperature and elasticity of the polymer. The classification of crosslinked polymers is based on the amount of crosslinking, spanning from lightly crosslinked polymers to highly crosslinked polymers to network polymers. These network polymers have crosslinked to the extent that all the chains have linked to form one large macromolecule.

The modulus of a polymer is tied to the flexibility of the polymer backbone. At a fixed temperature, this flexibility is reliant upon steric hindrance. All atoms, or groups of atoms, within a polymer occupy a certain amount of space, and steric hindrance is when the mobility or flexibility these groups of atoms is hindered by the presence of adjacent groups of atoms. Steric hindrance is dependent on multiple parameters, including bond length, bond rotation, the size of the functional groups, and polymer architecture.

Dielectric breakdown is the threshold at which an electrically insulative material forms a conductive path and fails to prevent electrical conduction through itself. Dielectric breakdown strength is the value, measured in field strength per unit length, at which dielectric breakdown occurs and is specific to each material. Dielectric breakdown usually begins with small leakage currents that eddy around and through defects. These small leakage currents generate localized warming, which increases the localized current, which increases the localized warming in an endless, degenerative cycle that eventually results in dielectric breakdown.

2.3 Linear Polymers and Plasticizer Technology

The first material class in consideration was linear polymers and their well-documented plasticizer additives technology. Plasticizers are the traditional polymer additive and are added to a resin to increase flexibility and plasticity. They have been developed for varying purposes: release agents and anti-blocking agents that migrate and concentrate at surfaces to prevent cohesion and adhesion, peptizers which chemically plasticize the material, processing and extender oils used as processing lubricants, and plasticizers which physically plasticize the material⁹. Plasticizers increase flexibility, or lower the modulus, and are not bonded to the material and effectively act as a lubricant between polymer chains; plasticizers work their way between chains, increasing the separation distance between the chains and their substituent groups and allowing the chains to slide past one another with decreased steric hindrance. Unfortunately, the primary strength of plasticizers is also their weakness; since plasticizers are not bonded within the material, they eventually leech out of the material returning its properties back to its former, pre-additive state.

Linear polymers embody a large portion of the polymer industry and have many applications ranging anywhere from structural to consumer products. Unfortunately, even with the addition of plasticizers, linear polymers are not capable of the large elastic deformations required the project criteria, deeming them unsuitable for use in the artificial muscle design.

2.4 Sylgard 184 Elastomer, by Dow Corning

Elastomers, a special class of networked polymers, receive their name for the rubber-like elasticity they exhibit. Elastomers were selected for the artificial muscle design due to their large elastic deformations and generally low moduli in compression. Sylgard 184 Elastomer, manufactured by Dow Corning, is a polydimethyl siloxane (PDMS)-based resin system and was chosen for its chemical structure, light-throughput characteristics, and high dielectric strength (Table I).

Table I. Typical properties of Sylgard 184 Elastomer, from Dow Corning.

Color	Clear
Viscosity	4,575 cp
Dielectric Strength	21 kV/mm
Curing Agent	87-RC
Mix Ratio	1:10

PDMS has a silicon-oxygen (Si-O) polymer backbone with methyl functional groups (CH₃) bonded to the silicon atoms in the backbone (Figure 5). PDMS has very little steric hindrance due to this chemical structure. The Si-O bond being recognized as one of

the longest bond lengths and the absence of covalent double or triple bonds, allowing bond rotation, lends itself to a flexible polymer backbone. Additionally, the lack of branching and only having small methyl functional groups prevents further steric hindrance. Perhaps most importantly, the oxygen in the backbone can only bond twice,

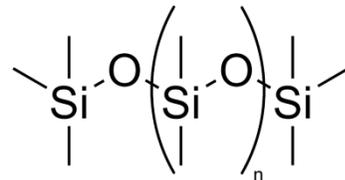


Figure 5. Chemical structure of polydimethyl siloxane. Methyl groups (CH₃) are implicitly located at the end of the bonds above and below silicon (Si).

leaving large spaces between functional groups and situating adjacent functional groups further apart.

However, plasticizer additives would be ineffective in elastomers because they lubricate the movement and sliding of polymer chains. Alternately, elastomers are networked and more of a flexible, rotating, and bending grid with no movement or sliding of chains. Instead, a hypothesis of disrupting the continuity of the crosslink density by adding a second, similar polymer with a longer crosslink length was adopted as the method to effectively decrease the modulus of compression.

2.5 3-6121 Low Temperature Elastomer, by Dow Corning

The viscosity of an uncured polymer resin is directly dependent upon the length of its prepolymer chains and chain architecture. Therefore, the viscosity of an uncured, PDMS, networked polymer resin, which has no branching or other architecture that increases steric hindrance, is coupled to the crosslink length. In choosing a secondary polymer for use as an additive, viscosity was substituted for crosslink length because the polymer industry does not publish specifics, such as crosslink length.

In order to decrease the possibility of adverse side reactions upon mixing, the secondary additive polymer was chosen from Dow Corning so any proprietary blends or additives would be similar. Additionally, the secondary additive polymer needed to use the same curing agent with the same mix ratio (Table II). This would decrease the possibility of the curing reaction completing at different rates, which could generate a material with

multiple phases. Finally, the secondary polymer additive required a higher viscosity than the primary material, Sylgard 184 Elastomer.

Table II. Typical properties for 3-6121 Low Temperature Elastomer, from Dow Corning.

Color	Translucent
Viscosity	19,250 cp
Dielectric Strength	18 kV/mm
Curing Agent	87-RC
Mix Ratio	1:10

3. Methodology

3.1 Spin Coating

3.1.1 Spin Coat Theory

Spin coating, a process commonly used in microfabrication to produce thin films of photo-resist polymer for use in etching silicon wafers, and can make thin films of any polymer with sufficiently low viscosity. The primary elastomer rests on the upper limit of this viability range, which causes problems to arise that will be discussed later in this report. As a circular substrate spins on the vacuum chuck within the machine, the centrifugal (outward) force on the polymer overwhelms the centripetal (inward) force put on the polymer by the substrate; extra material is expelled off the edge of the substrate (Figure 6).

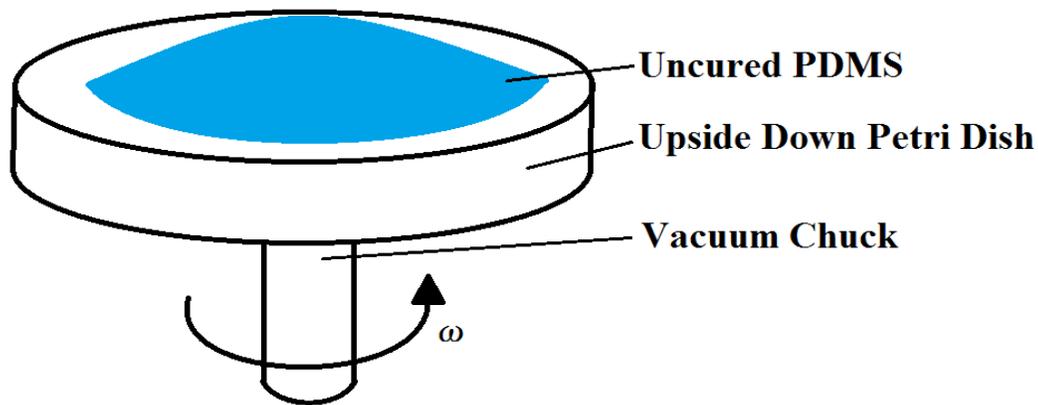


Figure 6. Cartoon of spin coater set up including dispensed PDMS

At higher spin speeds the centrifugal force expels more material which reduces the thickness of the resultant film. There is also an interaction between acceleration and spread steps within a spin cycle that can affect the final film thickness. Some detrimental effects on the resultant thin film will increase defect density if the wrong spin cycle is used, including streaking, uneven coverage, or microbubbles. This relationship is well documented for photo-resist polymers and is the basis for developing a spin cycle for use with PDMS¹⁰. An experiment was carried out that would allow for the characterization of a PDMS spin curve. The purpose of this curve is to attempt to reach a minimum film thickness possible by spin coating PDMS onto a polystyrene (PS) substrate. The resulting thin film is then cured in an oven and may require further processing in order to make accurate thickness measurements.

3.1.2 Experimental Procedure

The spin coating process utilized in this project entailed pouring uncured, mixed PDMS elastomer onto the underside smooth face of 80 mm PS Petri dishes, and spinning them at high speed for 40 seconds. The first 10 seconds comprised of an acceleration step at 300 rpm to allow the uncured elastomer to spread evenly over the substrate surface. The

second step lasted 30 seconds and utilized varying spin speeds to characterize a spin curve with increasing angular velocities. The spin speeds used ranged from 2000 to 6000 rpm. PS Petri dishes larger than 80 mm break when spun at the machines top speed, approximately 6000 rpm. These films were then cured in an oven for 1 hour at 70°C.

To make the film that will be tested in the actuation section of this report, a 100 mm silicon wafer was used for the substrate. An n-type wafer was used; however there is no difference between using n and p-type wafers. It was assumed that the difference in centripetal force between the elastomer and the two different substrates were negligible and that identical spin cycles would produce identical film thicknesses.

3.1.3 Mixing

The primary elastomer, Sylgard 184, was mixed from two parts, the resin and a curing agent. These components were mixed in a 10:1 ratio with 10 parts resin and 1 part curing agent. Mixing took place in various polyethylene cups containing a range of uncured elastomer volumes from 11 to 44 mL. These volumes were hand mixed with a stainless steel stir rod for approximately 1-2 minutes. Mixing ensures that the polymer experiences uniform crosslinking by distributing the curing agent throughout the resin. This introduces air into the uncured elastomer which must be removed before successful spin coating can occur. The work life of the primary elastomer is approximately 2 hours, before significant crosslinking raises the viscosity beyond that which is possible to spin.

3.1.4 Degassing

To remove the air introduced during mixing, cups full of uncured PDMS were placed inside a vacuum chamber. The reduced pressure within the chamber pulls the air from the uncured elastomer and all visible bubbles had been removed from the viscous polymer within 10-15 minutes. The chamber was then vented and the uncured elastomer was ready to be poured onto the Petri dish substrates and spun.

3.1.5 Measuring

Final film thickness was measured using an optical microscope with image capture software. In order to observe the edge profile of a film, samples were plasma bonded onto a slice of bulk PDMS elastomer that had been cast molded including micro-channels to make a distinction between the thin film and bulk PDMS. The thin film was then cut with a sharp razor blade to ensure a smooth edge and carefully peeled off each Petri dish by hand. Each film was measured three times and the film thickness was assumed to be the average of these measurements (Figure 7).

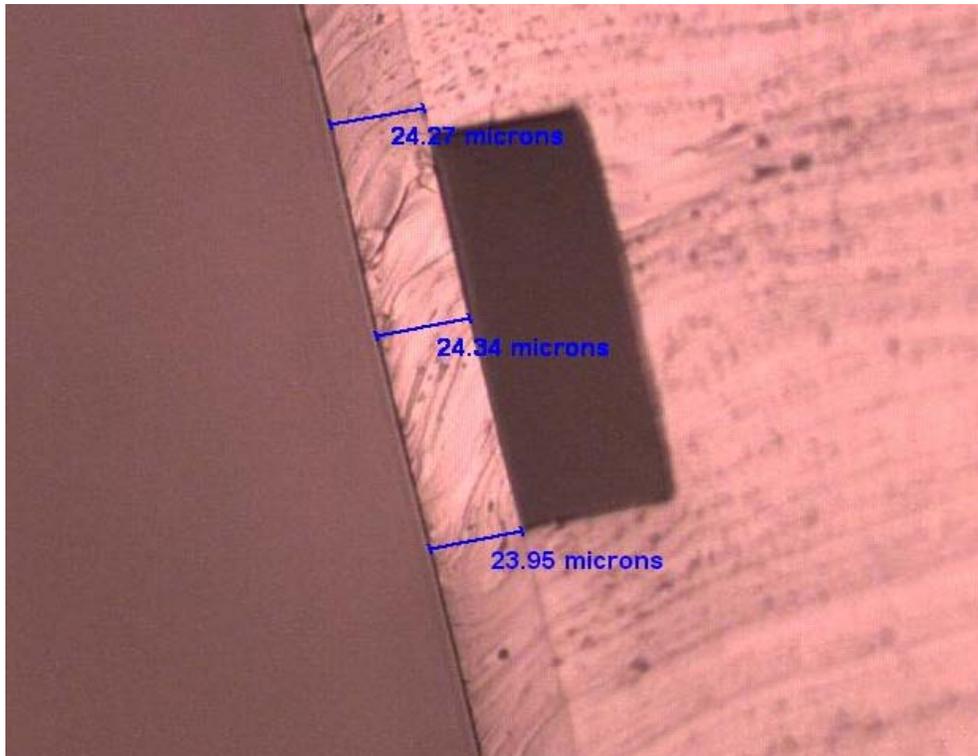


Figure 7. A sample micrograph at 200X magnification with bonded bulk PDMS showing a molded micro-channel.

3.2 Polymer Manipulation

In order to identify/categorize the compressive modulus of the Primary Elastomer and gauge the effect of using the secondary elastomer as a polymer additive, sample molds were created and samples were prepped with mixing, degassing, and curing. Finally, the samples were tested in compression on an Instron Tensile Machine.

3.2.1 Mold Design

Sample molds were necessary to ensure similar sample dimensions. Ten-millimeter sample molds were designed through the use of SolidWorks three-dimensional CAD software and prepared with an acrylonitrile butadiene styrene (ABS)-based polymer on a uPrint Rapid Prototype System, by Dimension, using a fused deposition modeling technique. Initial mold designs consisted of both a cylinder and a base with recessed

cylinder setting (Figure 8). After inserting the mixed, uncured polymer, a glass slide was laid across the top of the cylinder and then the sample was cured. During curing, the polymer would seep into the recessed cylinder setting making removal of the sample after curing difficult. The final mold design consisted solely of an ABS-printed cylinder with glass slides above and below (Figure 9). The resultant sample had an anvil height of $18.8\text{mm} \pm 1\text{mm}$ and an anvil diameter of $26\text{ mm} \pm 1\text{mm}$.

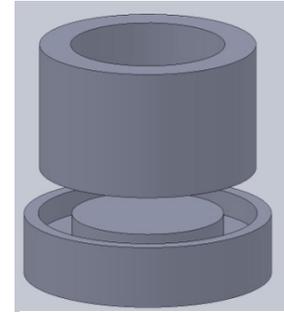


Figure 8. Preliminary compression sample mold, including base and cylinder.

The glass slides benefited the samples by creating a smooth and even surface on the top and bottom of the sample, but did lightly bond to the material upon curing, requiring the use of a razor to help dislodge the glass slide from the cured elastomer. The printed ABS cylinder did not bond with the cured elastomer, allowing the cured sample to slide out of the mold.

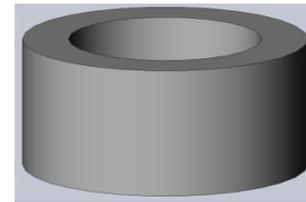


Figure 9. Final compression mold design. Glass slides were used as end caps for the cylinder when preparing samples.

3.2.2 Sample Preparation

The primary elastomer, Sylgard 184, and the secondary additive elastomer, 3-6121 Low Temperature Elastomer, both come as a two-part system, either part A/B or resin/hardener. Samples were created of pure primary elastomer, pure secondary elastomer, and compounds of the two varying volume percent. Samples were prepared in batches by first mixing the resins together via hand mixing for approximately two minutes, then adding the hardener at proper ratio to the entire volume of the resin and

hand mixing for approximately two minutes. Samples were degassed in the same method as the Spin Coating procedure (3.1.2 Experimental Procedure) and cured in a furnace for one hour at 70°C.

3.2.3 Compression Testing

For compression testing, an Instron Tensile Machine, model 3369, was fitted with a solid steel cylinder on the upper grip and a flat metal plate on the bottom grip. Samples were aligned beneath the upper cylinder on the base plate. The cylinder grip was lowered into contact with the sample and a slight preload was applied. After equalizing the load cell and extensometer, the samples were compressed at a rate of 10 mm/min up to 45% strain.

3.3 Actuation Characterization

The stress response to strain of the primary elastomer and an 80/20 mixture of Primary and 3-6121 Low Temperature Elastomer films were characterized. Interferometry was used to measure the amount of actuation that occurred when a stress was applied to the thin film. The PDMS elastomer was spun onto Si wafers to provide a reflective surface for the interferometer (3.1.2 Experimental Procedure). Stress was applied to the thin film via an electrostatic force and a mechanical force. For electrostatic testing an Indium Tin Oxide (ITO) coated slide was used as an electrode. This is because the ITO is optically transparent and allowed the thin films to be measured with the interferometer.

3.3.1 Interferometer Theory

Interferometry is a technique for measuring film thickness using light. The light from the interferometer is reflected back and the interferometer detects the interference pattern caused by the material. The thickness of the film is determined by comparing the maximum peaks within the interference spectrum. The two peaks were compared according to the film thickness equation (Eq. 2). As the film thickness decreases the distance between the peaks increase (Figure 10). The film and any object on top of the film must be clear in order for the interferometer to work.

Eq. (2)

$$t = \frac{1}{2 \left[\left(\frac{n}{\lambda_{1st \ max}} \right) - \left(\frac{n}{\lambda_{2nd \ max}} \right) \right]}$$

Where:

t = Film Thickness (nm)

n = Refractive Index

$\lambda_{1st \ max}$ = Wavelength at First Maximum (nm)

$\lambda_{2nd \ max}$ = Wavelength at Second Maximum (nm)

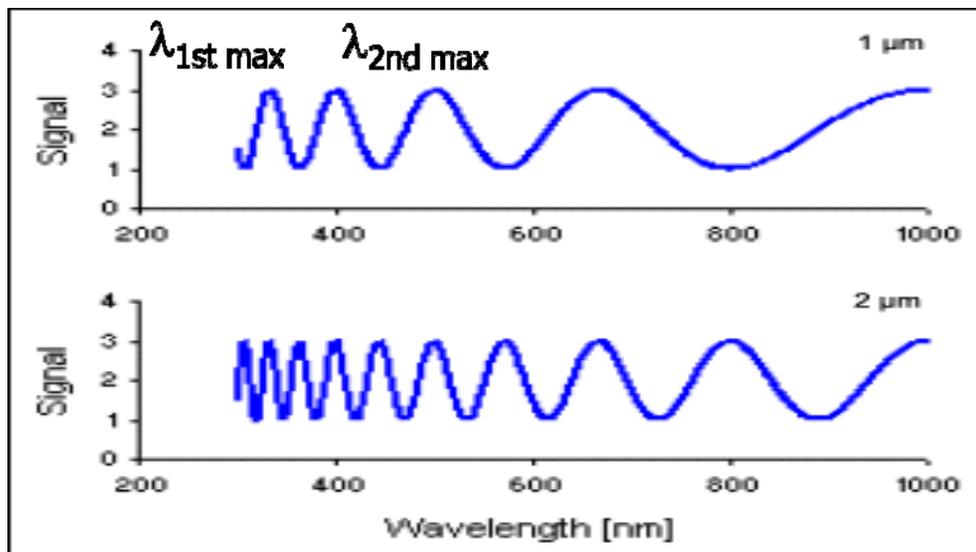


Figure 10. Typical Interference Spectrum showing change in distance between peaks.

3.3.2 Electrostatic

In the electrostatic actuation test, the thin film was placed between two electrodes and voltage was applied (Figure 11). The Si wafer substrate and a microscope slide coated with Indium Tin Oxide were used as the two electrodes. Voltage ranging from 500 V to 1600 V was applied in increments of 100 V. An initial base line of the film was measured at 0 V.

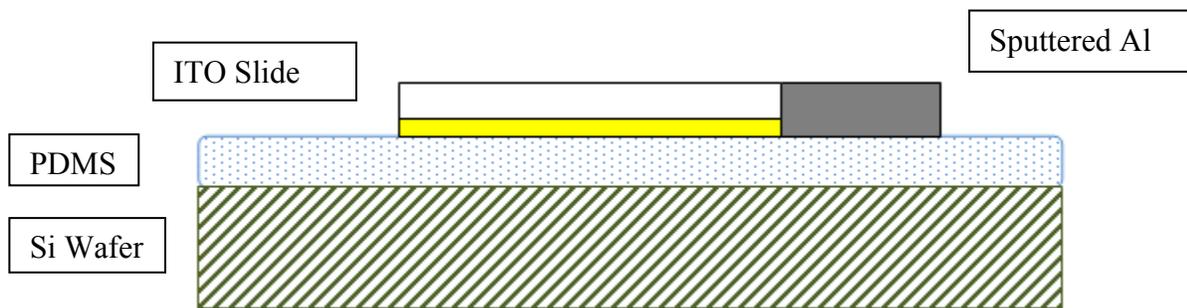


Figure 11. Electrostatic actuation cell with Al sputtered ITO slide.

3.3.3 Mechanical

In mechanical actuation testing, PDMS was placed under various weights (Figure 12). The weights used as mechanical force ranged from 0.5 kg to 2 kg in increments of 0.5 kg. An initial base line of the film was measured without any weights on it.

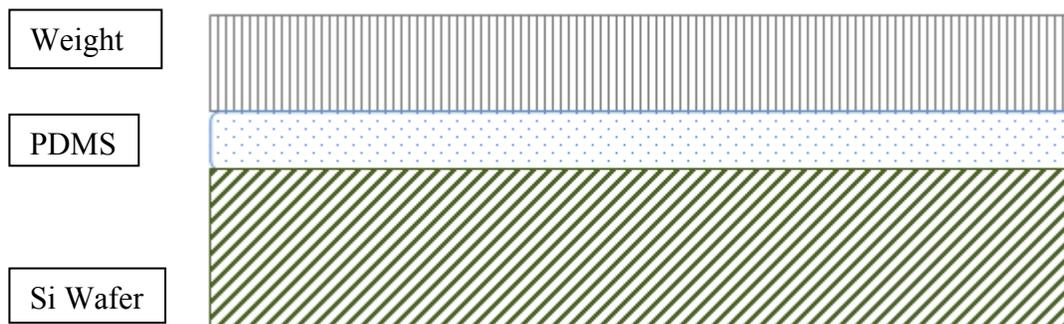


Figure 12. Mechanical actuation cell with mass weight.

4. Results & Analysis

4.1 Spin Coating

This single factor spin coating experiment included five levels of varying spin speeds with three replicates each. Graphing each replicate showed there was little variation between spin coating runs (Figure 13). The curve appears to reach an asymptote at approximately 15 microns. It is believed that this is the minimal film thickness possible with this elastomer using the spin coating method described above.

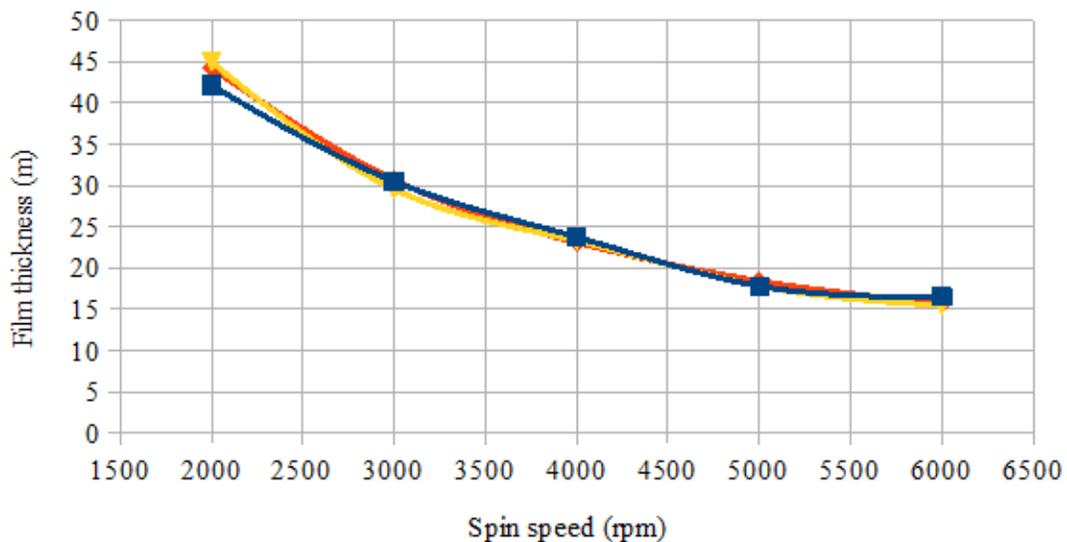


Figure 13. Spin curve characterizing PDMS on a PS substrate.

This is indicative of repeatable results, but the closeness of fit to a previously derived spin coat equation implies reproducible results as well. The spin coating equation is ordinarily used in microfabrication to predict film thickness of SU-8 photoresist polymers, but the viscosity of the primary elastomer is low enough that it can be modeled with the same equation (Eq. 3).

Eq. (3)

$$D = \left(\frac{4\rho\omega^2}{3\mu}t\right)^{-\frac{1}{2}}$$

Where:

D= film thickness (m)

ρ = polymer density

ω = angular velocity

μ = polymer viscosity

t= spin time (sec)

4.2 Polymer Manipulation

The polymer compounds, or mix ratios, tested varied by tens of volume percent and a total of nineteen sample compounds were created for compression testing (Table III). The distribution of samples across all compounds was not equal due to availability of the secondary additive elastomer, 3-6121 Low Temperature Elastomer. Large volumes of the additive elastomer could not be used, due to the nature of the elastomer to diffuse light, so the mix ratios with lesser secondary elastomer were favorable. Sample distribution was weighted towards the pure primary elastomer end of the scale, yet some samples were created of high volume secondary elastomer to characterize the effect compounding the two elastomers had on the compressive modulus.

Table III. Number of samples created with respect to volume ratio of Sylgard 184 to 3-6121 Low Temperature Elastomer.

% Sylgard 184 / % 3-6121 Low Temperature	#
100 / 0	3
90 / 10	2
80 / 20	2
70 / 30	2
60 / 40	2
50 / 50	2
40 / 60	3
30 / 70	1
20 / 80	0
10 / 90	0
0 / 100	2

4.2.1 Degassing

During the degassing step of the sample preparation, it was observed that the degassing time, the time required to remove all air from within the polymer, was dramatically shorter for the 80/20 compound than all other compounds. This identified that the 80/20 compound had an acute reduction in viscosity.

The samples of polymer compounds ranging from 30% secondary elastomer to 100% secondary elastomer did not degas completely and had air pockets caught within the material. The effect this incomplete degassing had on the compression testing is unknown, yet it would have caused many issues for electrostatic actuation testing and interferometry. Air bubbles diffuse light when contained within a material, so it is possible that the light throughput of some of these compounds would have rendered them

as viable options for electrostatic actuation testing via interferometry if the degassing had been complete.

4.2.2 Compression Testing

Samples exhibited a continuous, ever-changing compressive modulus that increased with increasing strain (Figure 14). It was also found that when a sample was tested a second time it produced results identical to those of its previous test. All samples exhibited similar compression curves, yet the curves differed in their slope.

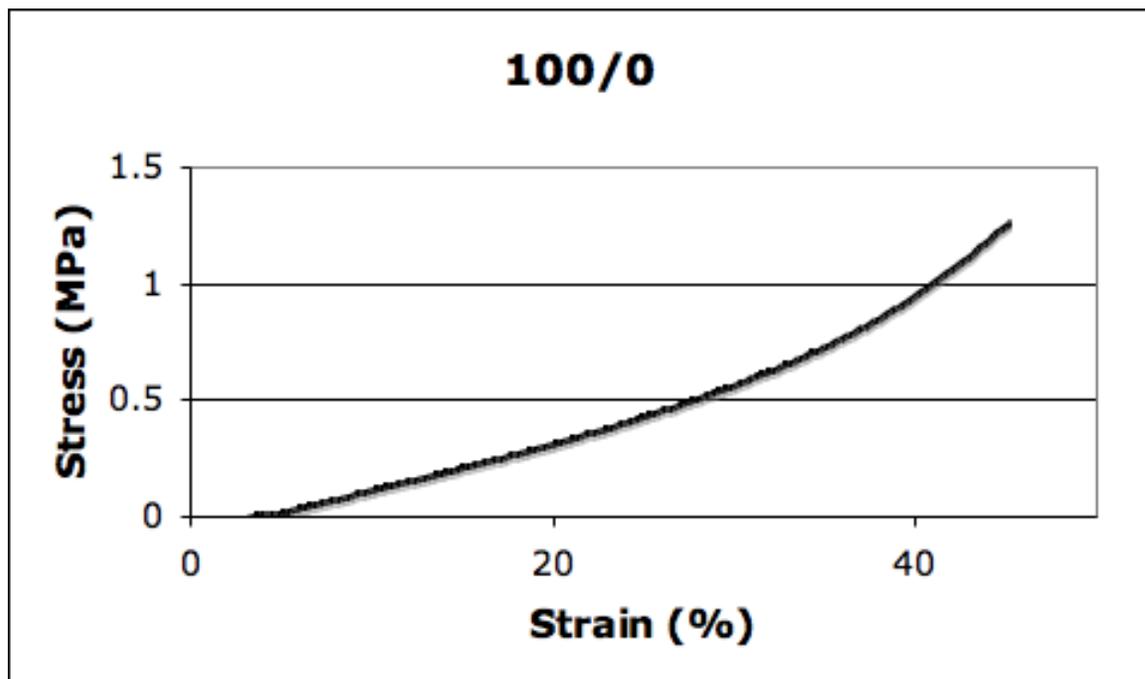


Figure 14. Stress v. strain graph of a sample of pure primary elastomer, Sylgard 184.

A continuous, compressive modulus presented a difficulty for drawing comparisons, so the stress response at a specific strain percent was substituted for the compressive modulus in order to draw comparisons. Comparing the average stress response at 20% strain across all compounds identified the 80/20 compound as having a dramatic reduction that challenged any possible trend (Figure 15).

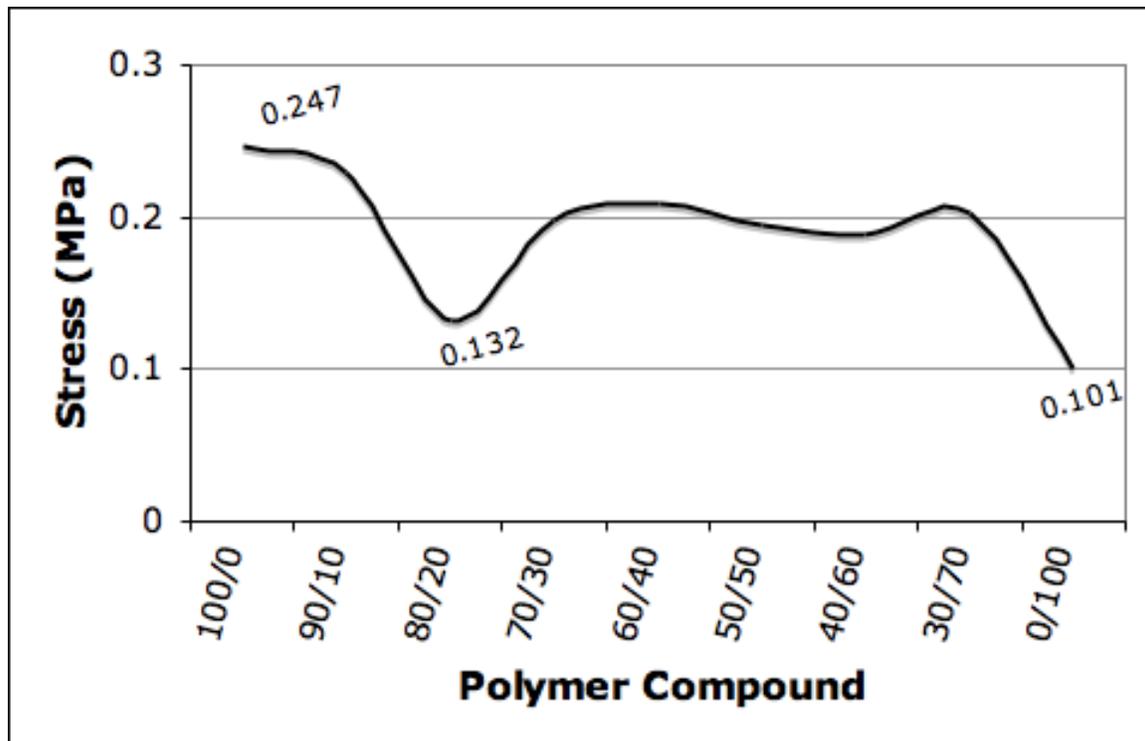


Figure 15. Graph depicting the average stress response across the varying compounds, with pure primary elastomer at the origin of the x-axis moving to pure secondary elastomer on the right end of the x-axis.

The compounds ranging from 30 vol. % to 70 vol. % secondary elastomer were found to statistically be no different than the pure primary elastomer, which had a stress response of 0.247 MPa. Both the 20 vol. % secondary elastomer compound and the pure secondary elastomer samples showed statistically significant differences in their stress responses of 0.132 MPa and 0.101 MPa, respectively.

A linear, decreasing trend from the high modulus, primary elastomer to the low modulus, secondary elastomer was expected, but the acute viscosity drop seen in the degassing did correlate into an acute decrease in compressive stress response, creating the nonlinear, average stress response across the varying polymer compounds. The unexpected results are likely a cause of error and there are three potential reasonings for where the error lies:

First, the 80/20 compound represents the ideal ratio of primary elastomer to secondary elastomer and there was no error, indicating that 20 vol% secondary elastomer was enough to disrupt the crosslink continuity and density without contributing to it.

Second, the incomplete degassing of the polymer compounds containing between 30 vol% and 100 vol% secondary elastomer caused the samples to exhibit an artificially high stress response; it is possible the stress response of these compounds should be similar to that of the 80/20 compound (Figure 16). The absence of tested compounds containing 80 vol% and 90 vol% secondary elastomer would help determine whether the drop from 70 vol% to 100 vol% secondary elastomer is more gradual or a drop such as that of the 80/20 compound.

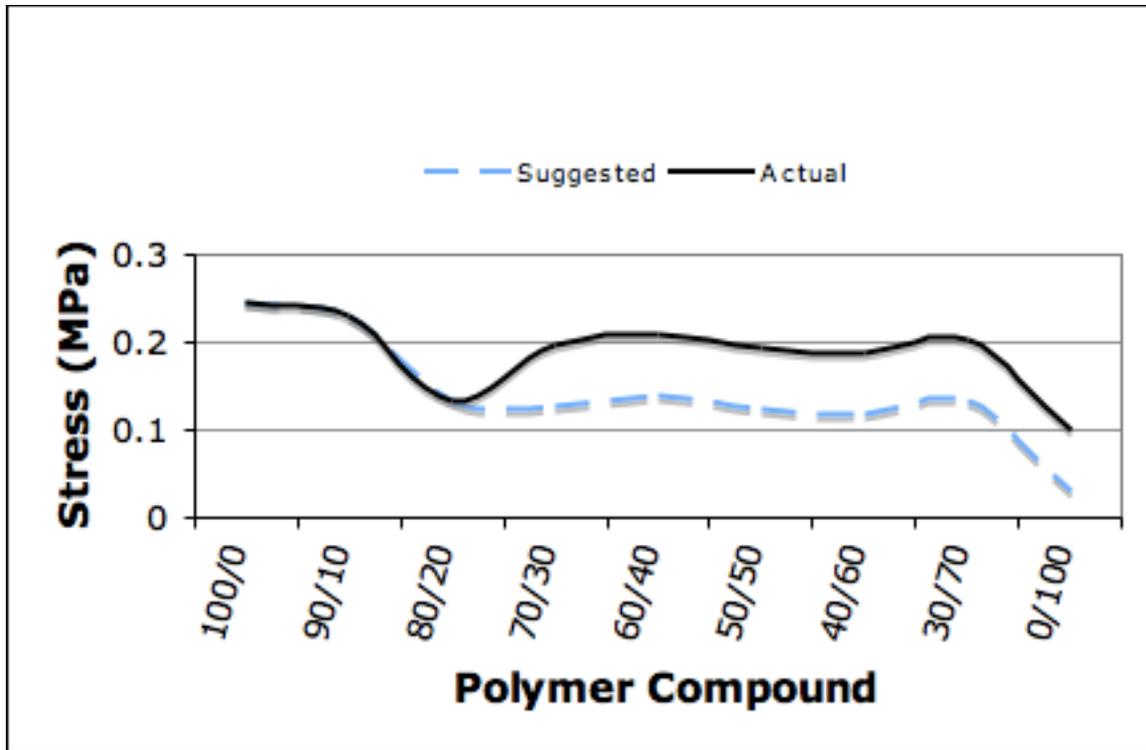


Figure 16. Graph depicting the actual average stress response across all the varying polymer compounds against a suggested average stress response according to the second reasoning.

Were the drop to 100 vol% secondary elastomer to be more gradual, the explanation that the samples with incomplete degassing exhibited an artificially high stress response would hold. The gradual decrease in stress response would indicate that the addition of the secondary elastomer did affect the stress response of the primary elastomer. Alternately, were the drop to 100 vol% secondary elastomer to be sudden, it would indicate the addition of secondary elastomer had little to no effect upon the stress response of the primary elastomer; the addition of the secondary elastomer either did not disrupt the crosslink density or the disruption did not affect the stress response of the primary elastomer, indicating that the acute drop in stress response for the 80/20 compound is artificially low and should be similar to the stress responses of the compounds on either side (Figure 17).

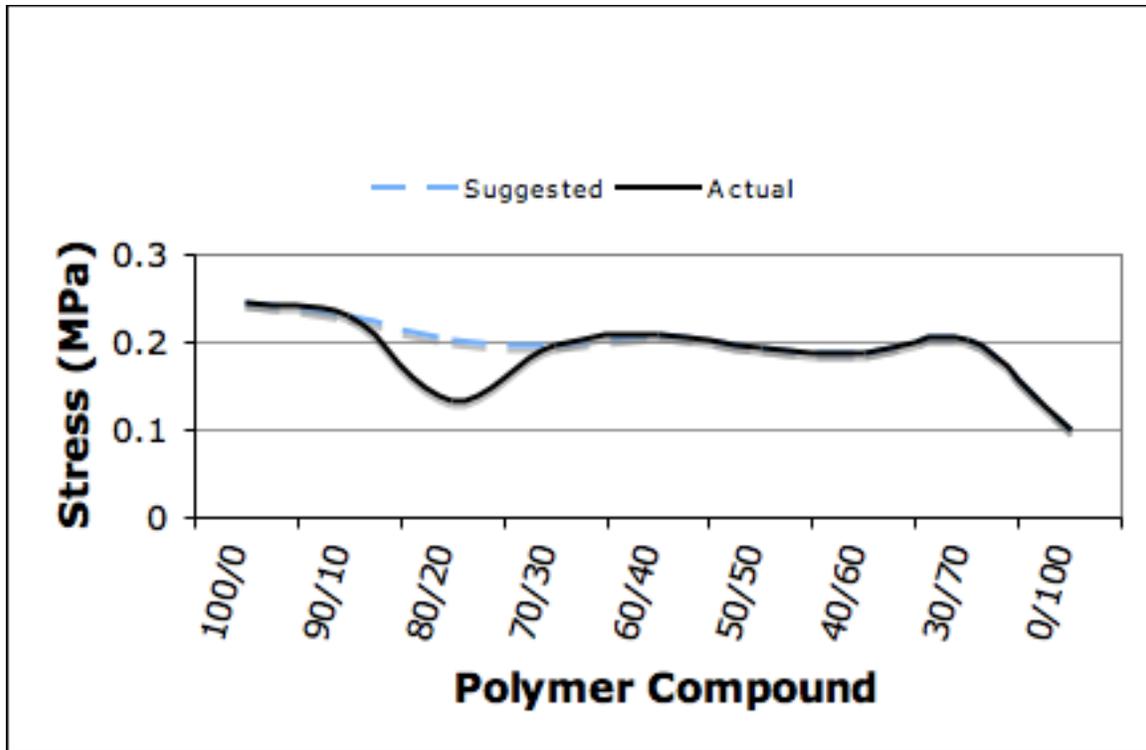


Figure 17. Graph depicting the actual average stress response across all the varying polymer compounds against a suggested average stress response according to the third reasoning.

Third, the stress response of the 80/20 compound is artificially low, either as the product of an uncontrolled variable or an error occurred in the sample preparation or a combination of the two. One such uncontrolled variable was the hand-mixing time in the sample preparation procedure (3.1.3 Mixing). Incomplete mixing can yield an uneven distribution of the curing agent throughout the resin, causing uneven rates of crosslinking. Such a scenario could produce an elastomer with highly-crosslinked portions and lightly-crosslinked portions, a multiphase elastomer. Alternately, if the hardener added was in excess of the hardener-to-resin ratio, the rate of crosslinking could have increased across the entire resin and possibly alter the method of crosslinking. Both the primary and secondary elastomers are highly crosslinked, but the functionality of neither elastomer is known. Were the rate of crosslinking to increase across the entire resin, it is possible that a tri-functional prepolymer, one that bonds three ways, would only have the opportunity

to bond twice, effectively becoming a bi-functional prepolymer, due to the lack of time for diffusion to occur.

4.3 Actuation Characterization

The actuation of the PDMS thin films was characterized using electrostatic and mechanical forces. The thin films were made out of pure primary elastomer and an 80/20 compound of primary and secondary elastomers. A 1600 V power supply was used for electrostatic testing, a set of weights ranging from .5 kg to 2 kg were used for mechanical testing and an interferometer was used to measure the relative film thickness.

4.3.1 Electrostatic

Voltages of 500 V to 1600 V were applied to the actuation cell. Both the pure primary elastomer and the 80/20 compound experienced dielectric breakdown between 700 V and 1100 V (Figure 18). Also, for both pure primary elastomer and the 80/20 compound, dielectric breakdown occurred at the point where the light from the interferometer was focused. The data obtained before failure yielded no actuation results.



Figure 18. Dielectric breakdown that occurred in PDMS thin film.

Dielectric breakdown occurs when an insulating material becomes conductive (2.2 Polymer Background). Current passes through the material and dielectric breakdown is observed. Dielectric breakdown occurred in the actuation cell due to defects within the film and surface contamination. Defects within the film were likely introduced during the degassing and spin coating process. It is possible the degasser was not able to remove all of the air bubbles within the PDMS. This resulted in voids within the film when the PDMS was spun onto the Si wafer. The surfaces of the PDMS films were contaminated with dust and other particles in the air during use. The number of defects within the film affected the voltage at which dielectric breakdown occurred. An increased number of defects and contamination causes the film to breakdown at a lower voltage. This is why the dielectric break down point within the PDMS films varied within 400 V.

4.3.1 Mechanical

Stresses from weights ranging from .5 kg to 2 kg were applied to the actuation cell. The thicknesses of the PDMS films were measured at each applied stress via interferometry.

The measured thicknesses of both the primary elastomer and the 80/20 compound were compared to the initial baseline measurement to obtain the percent strain of the film (Figure 19). The graph shows that the primary elastomer achieved a strain of 20% and the 80/20 compound achieved a strain of 35%. The primary elastomer experienced little strain until the 2.0 kg weight was applied. However the 80/20 compound experienced a large amount of strain when the 0.5 kg weight was applied and then remained constant.

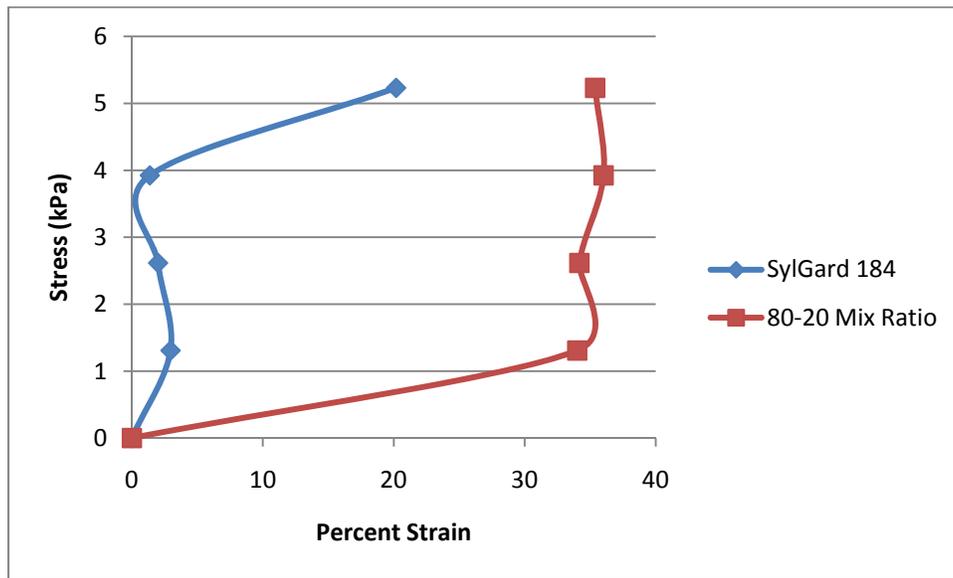


Figure 19. Stress-strain graph for actuation of PDMS films.

The difference in actuation shows that the 80/20 compound has more of a response to stress than the pure primary elastomer. The limit that the 80/20 compound had shows that there is possibly a maximum amount of actuation that the film can achieve, caused by the movement being restricted by the Si wafer. Also the 80/20 compound experienced a big jump in actuation before the primary elastomer. We believe that this is caused by a non-linear nature of the stress-strain response of elastomeric materials. It is possible that there is a steep, static modulus that transitions into a shallow, kinetic modulus. We believe that the 80/20 compound has this transition from static to kinetic modulus before the Primary PDMS which resulted in the 80/20 mixture having a large amount of actuation early on.

Only one test was completed for the actuation tests. This prevents the data from being verified through statistics. The outcome of the results could be the result of variation within the thin films. Uneven spread of the PDMS on the Si wafers could also interfere with the interference spectrum that the interferometer detects.

5. Conclusions & Recommendations

5.1 Spin Coating

Spin coating polymer thin films is a viable way to manufacture thin films down to a minimum thickness of 15 microns, yet care must be taken to reduce the probability of defects within the film. There is also little material loss and the results are highly repeatable and reproducible. Besides the cost of equipment, materials costs are minimal.

If defect density cannot be reduced to allow sufficient actuation by electrical stimulation, then a new method may be needed. One method to look into utilizes a stiff squeegee to remove excess material from microscopic thin molds. This method may have film uniformity issues which may only be solved through squeegee and mold dimension design. Both methods are promising in the cost-effective development of PDMS thin films.

5.2 Polymer Manipulation

This aspect of the project sought to decrease the compressive modulus of the primary elastomer, Sylgard 184, by disrupting the crosslink continuity and density through

compounding it with a secondary additive elastomer, 3-6121 Low Temperature Elastomer. Based on the observations of the sample preparation and the results of the compression tests, the following conclusions may be drawn:

- (1) Both the primary and secondary elastomers exhibited a continuous, kinetic compressive modulus where the stress response increased with increasing strain.
- (2) The compressive modulus does not change between trials of the same sample, making the results reliable.
- (3) The 80/20 polymer compound, 80 vol% primary and 20 vol% secondary elastomers, exhibited a drastic reduction in the compressive modulus, either as the result of error or precision.
- (4) Additional testing to characterize the transition from 70 vol% to 100 vol% secondary elastomer would help prove or disprove the assumption that the addition of the secondary elastomer affected the compressive modulus of the primary elastomer.
- (5) Using a centripetal compounder would thoroughly mix samples and circumvent the inaccuracy of hand mixing and the error originating from air inclusions within the cured polymer compounds.
- (6) The resin to hardener ratio should be included as a factor in future experimentation to account for any influence it may have had or might have on the compressive modulus of the polymer compounds.

5.3 Actuation

Actuation testing was performed to characterize the stress-strain response of the PDMS thin films. Electrostatic and mechanical testing was done. The results from the test conclude that:

- (1) Electrostatic testing failed due to dielectric break down caused by defects within the film and surface contamination.
- (2) The primary compound and the secondary compound were able to achieve an actuation between 20% and 40%.
- (3) The secondary compound achieved an actuation of 35%.
- (4) The primary compound achieved an actuation of 20%.

Future work should include:

- (1) A prestrain device to prestretch the PDMS films to allow for more actuation.
- (2) Running multiple tests to verify mechanical testing results.
- (3) Replication of mechanical testing results with electrostatic testing.

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