ACTIVELY MODIFYING TEXTURES USING SHAPE MEMORY MATERIALS

UNDERGRADUATE HONORS THESIS

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May 16, 2008

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ACKNOWLEDGEMENTS

First and foremost, I want to sincerely thank Dr. Marcelo Dapino for his continual support and guidance throughout this exciting and challenging project. I would also like to show appreciation of Neil Gardner for his assistance and advice provided in the student machine shop. In addition, I would like to thank Jeremy Seidt for his help in the running of the tension test experiments. I want to recognize Cameron Begg and Steve Bright at the Campus Electron Optics Facility for their assistance with the environmental scanning electron microscopy investigation. Finally, I want to express gratitude to the College of Engineering here at The Ohio State University for the Undergraduate Research scholarship and the general funding of my project.
This thesis presents techniques for modifying the surface texture of shape memory polymers with embedded active or passive wires. Previous work has looked at modifying the overall shape of shape memory composites. A key objective of this research is to achieve fully-reversible surface texture activation. The activated surface textures could be used for local aerodynamic control of automobile or aircraft panels by increasing local turbulence near the alterations. A turbulent boundary layer results in a smaller wake. Therefore, this new composite could be useful in applications where a material’s aerodynamic properties are carefully considered in the design process.
1. INTRODUCTION

1.1 Personal Motivation

Looking through the list of 2007 Honors project list created by Ohio State faculty, I was intrigued by one title “Actively Inducing Shapes and Textures” in the area of smart materials. At the time, I was unaware what smart materials were and did not have a clear idea of what the project entailed. During a meeting in March of 2007, Dr. Marcelo Dapino opened my eyes to the significance of the project. Dr. Dapino explained that the basic goal of this project was to alter surface shapes and textures using combinations of shape memory materials. Soon after Dr. Dapino and I mutually agreed on the project, I began a literature review on current studies regarding texture and shape modification using memory materials. I soon discovered that current research studies were looking at overall shape modification using these materials. Subsequently, I chose to take on the task of modifying textures on polymeric surfaces.

1.2 Industrial Significance

Currently, the automotive industry is exploring the use of shape memory materials as a method to reduce cost and to remove weight from vehicles. This is evident from shape memory polymer research currently being the single largest area of smart material research in the automotive industry based on the amount of patents filed. The result of this project is relevant in adaptable skin applications such as aerodynamic drag control of vehicles. Similar to a golf ball’s dimple, the modified polymer textures produced in this project will be able to actively create a local turbulent boundary layer associated with a lower overall drag coefficient. While the electrically heated shape memory material first order response may not be satisfactory for high performance racing, the materials have significant possibilities in the commercial market especially at elevated speeds on interstate highways. For example, as drivers enter an on-ramp to a highway, a new textured skin would form on the vehicle decreasing the cruising speed drag coefficient and saving on fuel economy on prolonged trips.
2. SHAPE MEMORY MATERIALS

Shape memory materials are subset of a larger collection of ‘smart’, or active, materials. Smart materials include certain types of alloys, polymers, and gels that can respond by changing material properties to various external stimuli such as light, magnetic fields, or heat. The low frequency bandwidth of shape memory materials results in a very slow response time and their stimulated output is a return to a ‘memorized’, or originally formed, state.

2.1 Shape Memory Alloys

Shape memory alloys (SMA) are thermo-mechanical materials that have high recoverable strain. The SMA memory effect is caused by two general microscopic forms, austenite and martensite. The SMA’s high temperature form, or austenite, has a body centered cubic structure while the lower temperature, or martensite, has a monoclinic lattice structure. When the atoms are cooled from the austenitic state, a mirroring effect called twinning occurs over a plane of symmetry. Detwinning occurs when a stress is applied to the material causing reorientation of the lattice structure. Heating the alloy will cause a non-destructive return to the austenitic lattice structure and the original shape will be retained. Figure 2-1 shows a flow chart of the alloy’s memory process. Martensite and austenite will coexist over the specific temperature region where cooling or heating takes place. The process exhibits hysteresis as shown in Figure 2-2.

Figure 2-1: Nitinol, a shape memory alloy, goes through a crystalline phase transformation in its memory cycle. Heating Nitinol realigns the alloy’s lattice into the austenitic cubic structure (top). Cooling the Nitinol creates a twinned monoclinic structure in the martensite form (left). The martensite form can be deformed and is realigned accordingly (right). Heating the alloy above the $A_s$ temperature returns it to the memorized cubic state.
First developed in 1962 at the Naval Ordinance Laboratory, Nickel-Titanium (NiTi) is one of the most important memory alloys due to its 10% strain recovery. The Nickel-Titanium Young’s modulus can increase 3 to 5 times when constrained and being heated from the martensitic to austenitic phase. Flexinol® is a commercially available NiTi actuator wire from Dynalloy, Inc. These wires act like muscle fibers when electrically driven. When heated, the wires contract which opposes the common idea of thermal expansion. This contraction can be up to 3-5%.

2.2 Shape Memory Polymers

Shape memory polymers are low cost, lightweight, and easily processed materials with a large strain recoverability of 400%. Under thermal stimuli, SMPs have the ability to “memorize” and recover an original formed shape. This memory effect is caused by a large difference in modulus below and above a specific glass transition temperature, \( T_g \). Unlike the crystalline phase transformation exhibited by shape memory alloys in response to heat, the SMP recoverability process is caused by a thermo-molecular relaxation. At the molecular level, SMPs involve cross-links, which determine a permanent shape, as well as switching segments that fix the temporary shape above \( T_g \).
A memorization cycle involves heating a rigid SMP thermoset above its \( T_g \) to induce a flexible elastic state. In this flexible condition, the material can be molded into an arbitrary shape and then cooled below \( T_g \) to retain this new shape. By reheating the SMP above the \( T_g \), the SMP will return to its original rigid shape by releasing stored mechanical energy attained during the reconfiguration and cooling of the material. This cycle can be repeated indefinitely without the loss of shape or degradation of the material. Figure 2-3 shows a Veriflex® SMP (Cornerstone Research Group, Dayton, Ohio).

![Veriflex® SMP](image)

Figure 2-3: CRG Veriflex® Shape Memory Polymer is a commercially available polymer manufactured in Dayton, Ohio. The memorized CRG Veriflex® SMP shape is the flat, rectangular plate (Top Left). As shown, the SMP can be heated, then twisted, rolled, stretched, or compressed (Top Right and Bottom). Cooling allows for the preservation of the new shape in a rigid state. Reheating these modified shapes above their glass transition temperature again will return the polymer to the memorized shape.

### 2.3 Shape Memory Composites

A shape memory composite (SMC) can be created from combining two or more materials involving at least one shape memory material. For example, one type of SMC could involve a shape memory polymer with embedded axial active or passive wires as shown in Figure 2-4.
Figure 2-4: A shape memory composite can be created by embedding active or passive wires in an SMP polymer. The wires can modify the composite’s stiffness, provide an internal heat source, or act as an alternative method of deformation.

The advantages of a shape memory composite are twofold. First, the addition of another material such as a wire can supply electrical resistive heat to the overall polymer matrix. Also, an embedded active wire can create a two-way reversible shape if specific austenitic finish and glass transitional temperatures are chosen. In this case of embedding an SMA into an SMP, the polymer’s memorized state can control the composite’s shape at lower temperatures. When the composite is heated, the softened elastic polymer would contort to the force of the austenitic wires. This relationship can be described in a Young’s modulus versus temperature plot shown in Figure 2-5.

Figure 2-5: Elastic Modulus versus Temperature plot for components of a shape memory composite.
3. FABRICATION

In general, the production of a shape memory polymer is a three part process. It is best to initially create a mold capable of containing a polymer resin as well as resisting raised temperatures during a cure cycle. Next, a two-part polymer resin must carefully be formulated to a specific consistency and transferred into the mold. Finally, the mold must be sealed from external air and must undergo a cure cycle at a raised temperature. If there is a desire to embed wires or a wire mesh into the polymer, the creation of a closed mold becomes more complex.

3.1 Creation of Molds

A mold is a container that allows a curable resin to form inside its cavity. Two distinct types of molds were explored in this project. The sheet glass mold was constructed from simple low-cost items. The second more complex ‘parabolic’ mold involved designing a custom ‘parabolic’ mold using a computer numerically controlled, or CNC, mill.

3.1.1 Sheet Glass Closed Mold

The first and most successful mold type explored in this project is a simple sheet glass mold. This type of mold is used to create a rigid flat sheet of polymer from a liquid resin mixture. The final polymer length, width, and thickness are determined by the dimensions of the sheet glass mold. The sheet glass mold is fabricated from five resources including sheet glass, Viton cord, binder clips, Viton or silicone epoxy, and mold release.

There are several advantages of using glass for the mold walls. First and foremost, the transparency of glass allows one to see the inner-workings of the resin accumulation during the filling stage of the mold. In addition, glass has natural chemically resistive properties and a low coefficient of thermal expansion. Finally, glass is cheap and readily available at local hardware stores where it can be cut to size.
Two glass sheets of equal size create the walls to form the wide faces of the polymer. The thickness of the glass was found to work in the range of 0.1 to 0.125 inches. There is no reason to suspect the glass would fail at increased thicknesses, but going below 0.1 inches may increase the risk of the glass sheet fracturing during polymer removal. The glass sheets were tested in this experiment at lengths and widths ranging from 3 inches by 4 inches all the way to 8 inches by 10 inches. The length and width are arbitrary.

The glass sheets are preferably cleaned with acetone prior to fabrication but water generally will suffice to clear residue from the glass surface. After the two selected equally sized glass sheets were cleansed, mold release is applied to the sides of the glass that will act as the inner cavity of the mold. Mold release keeps the viscous resin from attaching to the glass surface. In this project, the mold release that proved to give the optimal surface finish and least adhesion to the glass walls was CRG Industries Mold Release. The mold release is applied to the surface of the glass with a paper towel making sure to gloss the entire surface. The mold release is allowed to dry for five minutes and then the process was repeated. In all, three treatments of mold release were applied. Figure 3-1 shows an application of mold release.

Figure 3-1: Mold release is applied to the glass sheet walls of the polymer’s mold with a damp paper towel. Three applications are applied waiting five minutes between each treatment.
The next step in mold creation was to create side walls by placing a chemically resistant Viton® cord between parallel glass sheets. While the sheets of glass had a relationship with the surface area size of the cured polymer, the Viton® cord diameter defined the specific thickness of the polymer. The values of cord diameters tested in this project included 0.063, 0.094, and 0.118 inches (1.6, 2.4, 3 millimeters). CRG suggests not exceeding 0.5 inches in cord diameter. In general, it is easier to work with larger diameters of cords, but in this project the smallest diameter is more desirable in creating a thinner polymer specimen. The length of the cord is suggested to be equal to the perimeter of a glass sheet.

Figure 3-2: A Viton® cord is sandwiched between the two glass sidewalls. This is a necessary first step in creating a leak-proof mold. Once the cord is aligned a linearly along the bottom of the sheet, binder clips are clamped through the center of the cord on the outside of the glass.

The bottom of the glass mold should be decided upon before sandwiching the Viton® cord as this is the area that will have to support most of the resin weight. It was found best to orient the mold so that the bottom of the mold is of lesser dimensions than the sides so the mold looks like an upright rectangle. As pictured in Figure 3-2, the cord is positioned linearly across the bottom having the distance of a selected binder clip’s ‘reach’ away from the bottom of the glass. In general, a ¾” binder clip refers to having 0.75 inches of reach. Next, the second sheet of glass should be placed upon the top surface of the cord effectively sandwiching it into place. Binder clips are clamped over the outside surfaces of the glass making sure to apply the clamping force onto the cord. Figure 3-3 shows the clamping of the Viton cord. Once the bottom of the mold is completed and the cords are held securely in place, the cords are wedged between openings on
the sides of the mold and clamped similarly to the bottom of the mold. In repeating the wedging of the cords on the top of the mold, it is important to leave a gap for the pouring of resin.

![Figure 3-3: After completing the bottom wall, the mold’s side walls are created using Viton® cords.](image)

After the mold is filled, the gap can be closed by touching two opposite ends of the cords, dabbing a seal of silicone or Viton® epoxy between the touching ends, and clamping the remaining cord. It is very important to make sure the resin is effectively sealed. The resin contains volatiles that will be driven out of the resin during the cure if it is not contained in such an airtight mold. The resin will also oxidize severely in the presence of oxygen. Either condition will leave a very unsatisfactory polymer.

### 3.1.2 Polymer Parabolic Closed Mold

An exploration was made to determine the possibility of custom shaped molds. A custom shaped mold has the advantage of creating polymers with decidedly different shapes. As for reasons explained later in the report, a decision was made to cure a parabolic polymer bump after creating numerous polymer sheets. It was decided that the mold to create such a polymer would have to be created using a CNC mill. A few concept sketches led to a CAD model of both a top and bottom mold drawings as pictured in Figure 3-4 and Figure 3-5 respectively.
Figure 3-4: Top of the Parabolic Mold

Figure 3-5: Bottom of the Parabolic Mold
Figure 3-6: The parabolic mold assembly shows a 2-dimensional cross-section of the curable area of the custom mold. A polymer bump that is 0.040 inches in thickness will be the end result.

The purpose of the mold is to fill a 0.040 inch gap (indicative of the red area in Figure 3-6) between the concave and convex plates with resin and mate them together to form a closed mold. The CAD model is loaded into FeatureCAM which allowed for the specification of CNC milling techniques. After testing the process in wood blocks, the two plates are machined out of Lexan polycarbonate sheet for its transparency, high working temperature, machinability, and chemical resistant properties. Similar to the sheet glass mold, the inner walls of the parabolic mold are glossed with mold release to prepare it for the resin filling.

### 3.2 Polymer Creation

The second step in the fabrication of a polymer involves creating a specific resin to fill the closed mold. Veriflex®, a two part resin mixture of choice, is available commercially from Cornerstone Research Group Industries.
3.2.1 Resin Mixture

A CRG two part resin mixture is used to create the Veriflex® polymer. Part A is Veriflex® resin while Part B is a hardener that was added to activate the resin. The mixture is added on a volume basis where the final solution consists of 96.9% Part A and 3.1% Part B. The simplest way to get the exact amounts required is to work on a mass basis. A clean glass dish or aluminum foil lined plastic dish is tared on a scale to account for the unladen weight. An estimated amount of Part A capable of filling a finished mold is poured into the dish and its mass is measured. The following equation determines the mass of Part B to add that gives the necessary volume percents.

\[
\text{Part A Volume [mL]} = \frac{\text{Part A Mass [g]} \times 313 [mL]}{288 [g]}
\]

\[
\text{Total Mixture [mL]} = \text{Part A Volume [mL]} \div 0.969
\]

\[
\text{Part B Volume [mL]} = \text{Total Mixture [mL]} - \text{Part A Volume [mL]}
\]

\[
\text{Part B Mass [g]} = \frac{\text{Part B Volume [mL]} \times 12 [g]}{10 [mL]}
\]

After combining the correct masses of Part A and B, the mixture is stirred until all white hardener is evenly distributed within the clear resin as pictured in Figure 3-7. Gentle stirring prevents the production of large air bubbles. Once created, the resin is very viscous and resembles the consistency of maple syrup. Chemically resistant gloves are necessary due to accidental spills and leaks. Also, the resin is worked with under a fume hood to negate the effects of the mixture’s intense chemical smell.
3.2.2 Filling Closed Molds with Resin

Filling a mold can be done in a few different methods. CRG suggests using a glass syringe to transfer the resin into the open cavity at the top of the mold. The small width of openings (e.g. 0.063”) makes it difficult to find a small enough glass syringe that pumps the viscous resin at a reasonable rate. Attempts were made to use plastic syringes, but the caustic resin soon began to soften the walls of the syringe and it became unusable.
The best method discovered to both allow for a relatively quick and clean resin fill is to create a small aluminum foil guide to channel the resin into the mold as shown in Figure 3-8. The guide is created by folding a 2 by 3 inch piece of aluminum foil in half to create a v-shape. The concave side of the ‘V’ is loaded with small amounts of resin with a glass or metal scoop. Then, carefully, the guide is tilted into the opening on the top of the mold. Generally, a wider opening allows to easily and quickly fill the mold, although it takes a large volume of resin. A typical resin accumulation is pictured in Figure 3-9. At the 0.063” width, it is important to pour very slowly and to make sure there is no overspill running down the sides of the mold. It is also important to look for any leaks of polymer where the Viton® cords are clipped. In addition, a small empty volume was left above the settled resin to allow bubbles in the resin to rise. In general, all of the large bubbles that form during the pouring stage rise to the surface and escape from the resin. Some very small bubbles remain, but tend to dissipate during the curing phase.

![Figure 3-9: Resin slowly fills the mold’s volume. This is an important time to check for leaks in the mold.](image)

The filling of the parabolic closed mold is more difficult for a few reasons. To begin with, it is very difficult to move the viscous resin into the small 0.040 inch mold cavity. The first attempt at a mold filling technique was to drill a small hole in the top of the top mold and to pour the resin into the opening. This was an attempt to let gravity do the filling work as accomplished with the sheet glass molds. The resin ultimately failed to run down all of the sides of the
parabola and formed large bubbles that could not escape from such a small air hole. More attempts were made with this filling technique including adding more air holes to allow bubbles to escape, but ultimately they failed as well.

A simpler sandwiching technique was devised. This technique involves filling the concavity of the top mold with resin and slowly twisting the convex mold piece into place essentially spreading the polymer into all available mold cavity space. This method also has some problems as discussed in Section 4.3.1.

### 3.2.3 Polymer Cure Cycle

To turn the viscous resin into a rigid polymer, the mold has to undergo a curing cycle. CRG Industries recommends two unique curing cycles. Both cycles need to cure vertically as pictured in Figure 3-10. The first attempted style of the two methods is a shorter but more involved cure cycle. This cycle involved 4 steps:

1.) Ramp from room temperature to 75°C with a heating rate of 1°C/minute.
2.) Soak at 75°C for 3 hours.
3.) Ramp from 75°C to 90°C with a heating rate of 15°C/180 minutes.
4.) Ramp from 90°C to 110°C with a heating rate of 20°C/120 minutes.

Although this process produces a rigid polymer more quickly, the 10 hours of following instructions became tiresome after the first few cures. Also, this process allows more error to enter into the cure cycle due to the low resolution dial temperature input and analog temperature display.
The second attempted method proved to be much more successful. This process involves preheating the oven to 75°C and curing the mold for 36 hours at this temperature. Although this cycle takes 3.6 times as long as the first method, there is virtually no work involved once the polymer has begun the process. Occasionally it is advised to check on the mold to make sure no problems arise such as oxidization or a slow leak. Also, as noted by a CRG Industry contact, the 36 hour cycle tends to reduce cure shrinkage. At the end of both of the cure cycles, the oven is turned off and the cured polymer is allowed to cool inside the mold.

### 3.2.4 Removal from Mold

After the polymer has cooled, the next step is to remove the polymer from the mold. The first removal step is to unclamp and set aside all of the binder clips. Some binder clips may stick to exposed epoxy or small amounts of over spilled resin. These binder clips are simply loosened with any type of sharp slender tool such as a knife and then removed. Despite the application of mold release, the polymer still adheres to the mold release covered glass after the binder clips are removed. A wedge device is used to separate the glass sheets from the polymer. It is important to work delicately when wedging apart the glass due to its tendency to fracture if too much pressure is applied. Working around the mold in a circular pattern proved to work best in loosening the polymer’s grip. Small natural “popping” sounds occur when the polymer separates from the glass. The separated area becomes visible when looking at the polymer through the
glass. Once most of the polymer is separated it generally releases from one glass sheet. A knife is used to wedge the polymer from the remaining glass.

3.3 Wire and Mesh Embedding Techniques

For reasons explained in Section 2.3, there is a desire to embed wires or wire mesh into polymers to create a composite. Active Flexinol® wires ranging from 0.003” to 0.020” in diameter were embedded in the polymer during the initial phase of the project. Later on, passive resistive copper and aluminum wire meshes were inserted into polymers.

These new metal additions create a significant mold design problem for a few reasons. First, the wires optimally need to be centered in the thickness of the polymer. Secondly, the wires need to stick out of the polymer to act as lead wires for an electrical circuit. Finally, the wire material should not chemically react with the volatile polymer resin as early tests discovered.

The glass sheet mold was chosen as the best option in creating the composite mold due to its success in creating polymers without wires. Working within these mold restrictions, there is not a natural way to center the wires in the polymers between 0.063” to 0.118” widths. The first attempted idea was to string wires through the width of the polymer using the Viton cords to clamp them into place. Although this approach is not optimal, Figure 3-11 shows the diagonal method of approach. This method involves the difficult task of keeping the wires taut especially at smaller diameters. It is best to weigh down the end of the wires and then to clamp them in this stretched position. The process is verified in a few of the initial composites but an easier method is still desirable.
The next attempt at centering the wires involves using a slender pin to poke holes through Viton cords. Then wires are punched through these small openings. In early testing, this process proved to only work with larger diameters of Viton cords and wires. The larger Viton cords (0.118”) are much easier to poke a hole through without degrading the structure of the rubber. Smaller diameters would sometimes split in the process of poking holes with a pin. Once the wires are strung through opposing sides of the cords, the Viton naturally seals the small gaps in the pin holes under the clamping pressure. Also, larger diameters of wire are easier to poke through the small openings due to their stiffness. Figure 3-12 shows a diagram of how the wires were placed in the mold.

For reasons discussed in Section 4.3.3, the project began to require an embedded wire mesh. Three types of copper mesh as well as one aluminum mesh were investigated. Initially, a method similar to the diagonal wire method was attempted. Unlike the small cross-sectional areas of the wires, the cross-sectional area of the mesh proves to be large enough to displace a decent amount of Viton cord and thus presents a resin leakage problem. This leakage cannot be easily fixed.
with sealant due to the sealant’s disintegration when in contact with the resin mixture. Therefore, the mesh needs to be contained entirely within the mold with no leads sticking out.

In theory, a second technique solves the containment and centering problems. This method involves creating a square cutout of mesh between 1 to 2 inches. The mesh is inserted into slots that are cut into small Viton cord sections. Figure 3-13 shows the theoretical idea of using these Viton ‘clips’ to center the mesh inside the mold’s thickness. Figure 3-14 shows a copper mesh being held between the glass sheets using just the clamping force of the binder clips to hold its vertical position.

![Figure 3-13: A cross-section of a Viton ‘Clip’ held mesh shows the ease of centering with this method.](image)

The Viton clips also solve the containment of the electrical connections. The idea is to cure a polymer with the clips holding the mesh in place. The Viton clips can then be pulled from the
polymer sheet once it is removed from the mold. This removal exposes a section of open wire mesh. These open wires can be used to establish an electrical connection on opposing sides of the polymer. The cured polymer in Figure 3-15 shows a Viton clip cured into place.

Figure 3-15: A zoom-in on Viton ‘Clips’ in Cured Polymer Sheet shows that if the clips are removed properly, wire leads will be exposed.

In practice, the removal of the Viton clips does not work straightforwardly. Often, small amounts of the resin leaked in between the clips compromising the wire leads. Also, the removal of the clips from the polymer regularly damaged the delicate wire lattice by pulling small wires loose.

Another general failure of the initial mesh embedded polymers involves the choice of wire material. As mentioned previously, the volatile resin is capable of reacting with many materials. The Flexinol® wires (NiTi based) shows no reaction to the volatility but the first copper mesh molds presents a clear demonstration of an oxidation reaction. The polystyrene based resin reacts with the copper to form cupric salt crystals in the cured polymer as shown in Figure 3-16. Clearly, these copper meshes are not be useable. To show another example of resin’s volatility, Figure 3-17 shows a reaction with common paperclips.
Figure 3-16: A corrosive reaction between the copper mesh and polystyrene polymer resin creates a cupric salt product.

Figure 3-17: A nickel plated paper-clip reacts in the Veriflex® Resin. Veriflex® resin is very volatile and may corrode many embedded materials.
Fortunately, simple aluminum screen mesh allows for the creation of high-quality shape memory composites. The aluminum screen mesh has many advantages over its copper counterpart. Compared to the copper, the screen mesh is more flexible, more electrically resistive, and available for free at the mechanical engineering department’s student machine shop. The best feature is its corrosion resistance to the resin.

The final and best practice of embedding meshes in the polymer again solves the issues of centering and containment, but this time it does so in practice. The idea is to contain the lead connections inside the mold by running them both vertically out of the polymer resin into the open cavity remaining above the polymer fill. Various shapes of resistive heating networks were cut from the screen mesh but they were all required to leave to sections of mesh protruding the surface of the polymer as shown in Figure 3-18.

![Figure 3-18: A ‘U’ shaped aluminum wire mesh is embedded into the polymer. Protruding lead connections are necessary to obtain a internal resistive heating network.](image)

This protrusion of the lead connections requires a small modification in the resin fill process. To limit the amount of resin stuck in electrical connections, the pouring of the resin is limited to the areas where no metal would protrude from the surface. This means pouring near the Viton® cord side walls and in between the connections as shown in Figure 3-19. There is an extra amount of time required for the slow moving resin to settle inside the mesh apertures and to allow bubbles to escape. In addition, the mesh is naturally held near the center of the mold thickness by opposing viscous forces in all cured polymers using this method.
Figure 3-19: Resin slides down the cavity of the mold between the electrical leads. It is important to keep the resin away from the leads so proper electrical connections can be achieved.
4. CHARACTERIZATION AND RESULTS

4.1 Validation of Polymer Properties

Figure 4-1 shows the first cured polymer in April of 2007. This early polymer is milky white and has a rough surface finish due to the first type of spray-on mold release used. This opaque dry-coat mold release was quickly discarded for transparent spray-on mold release. The results were improved but the polymer still was slightly milky in color. The final step towards a perfect polymer involved switching to a mold release provided by CRG. Once the ideal polymer was repeatable, polymer properties were tested numerically in tension tests and observationally by examining a memory cycle.

Figure 4-1: The first cured polymer was barely transparent and had a rough surface finish. Early polymers were treated with spray on polyurethane mold release and underwent a 10 hour cure cycle.
Figure 4-2: The perfected polymer was transparent having a smooth surface finish. Improvements included using a mold release provided by CRG as well as a 36 hour cure cycle.

4.1.1 Tension Tests

Tension tests were used as a numerical means of validating the cured polymer’s properties versus CRG’s provided property data. The polymer tensile test method was run according to ASTM D638 standard and was tested on an Instron testing machine. This standard offers five approved dumbbell sizes of which Type V is most favorable due to its small dimension shown in Figure 4-3. A CNC milling process was used to cut the Type V dumbbells from a polymer sheet. The CNC process allows for excellent accuracy and smoothness in the cutting of the dumbbell gage length dimensions.

Figure 4-3: An ASTM D638 ‘Type V’ Dumbbell was used in tension tests to verify material strength properties. The dimensions are given in inches.
For this Type V test, test grips had to be obtained. A few manufacturers of tensile grips were contacted, but the prices of the grips were unreasonable for the small amount of testing time desired. Instead, it was decided that a custom grip would be built in the student machine shop. The grip is designed as shown in Figure 4-4.

![Figure 4-4: A custom tensile grip design was developed to attach to an Instron tensile test machine with a threaded attachment. The dimensions are provided in inches.](image)

The tensile testing grips are machined from aluminum bar stock for this specific dumbbell size. A milled slot is used to grip the specimen. TRA-BOND 2106T epoxy is applied to both the surface of the slots as well as the grip areas on the dumbbell. A 24 hour epoxy cure process allows the test specimen to congeal into place. After this, the testing grips are then attached to the Instron testing machine via left-hand thread connection to the actuator and a right hand thread connection to the load cell. The process of threading the grips into place is a delicate process. One man turns the grips and the attached specimen while one man controls the raising of the actuator. It was found that raising the actuator at a rate of 0.002 in/s was a good speed. An Epsilon Model 2442 extensometer is placed on a region where the significant lengthening of the gage length was expected to occur as shown in Figure 4-5.
Figure 4-5: An extensometer was attached to gage length of the dumbbell. A returned voltage signal could then be converted to change in length in test analysis.

The results of the testing proved that the polymer specimen’s tensile strength value was within a small error of CRG’s data. As shown in Figure 4-6, the tensile strength of the polymer was found to be 3237 psi while CRG’s data showed 3300 psi. It was decided that 1.9% error was close enough for the purposes of this project.
4.1.2 Memorization Cycle Tests

While the tensile properties of the polymer had been validated, there was necessary testing to discover whether the cured polymer could demonstrate the memorization cycle. A simple experiment was started by cutting a four inch by one inch plate of polymer that was 0.094 inches in thickness. This plate was clamped in an upright position and was subsequently heated with a heat gun until the entire surface area had a temperature above 62°C. The temperature was checked by placing thermocouple wires onto the surface of the polymer. Once it surpasses this $T_g$, the polymer became elastic to the touch. The heat source was then removed and the polymer was bent and twisted into a new shape and held there until the polymer once again became rigid. The memorization cycle was proven by reheating the polymer past $T_g$ and discovering that the polymer unbent and untwisted itself towards its original position. The memory process quickly took effect once the polymer became soft again.
4.2 Shape Memory Composite Results

In this project, both active wires and passive wire meshes were embedded into active polymers creating shape memory composites. These wires are added to act as a resistive heating network and an alternative means of modifying a polymer.

4.2.1 Axially Loaded Polymer Results

The first attempted shape memory composite involves embedding Flexinol® wires through the length of a sheet of Veriflex® polymer. The goal of this composite is to provide an axial force capable of causing an ‘accordion’ effect on the polymers surface. An electrical current induced through Flexinol® wires causes the wires to contract. In this contraction, the accordion effect would be caused by the friction, or bond, between the wire diameter and the surrounding polymer. The desire is for the accordion, or ripple effect, on the surface of the polymer to be perceptible in touch and sight. Various diameters of wires ranging from 0.003 to 0.020 inches were tested for their surface texture alteration. The larger diameter wires offer a greater contracting force and were suspected to have a much larger surface effect.

Figure 4-7 shows the idea behind the axial loaded polymer as a five step process. Step 1 represents a rigid polymer with unheated axial Flexinol® wires. In Step 2, the wires are heated above their austenitic finish temperature and contract up to 5%. This causes the composite to contract in as well due to frictional bonds and small expansion of the wire diameter creating the surface ripple effect. The wires are cooled to their martensitic state. This leaves the polymer in its deformed state as shown in Step 3. In Step 4, the wires are reheated to warm the polymer past the glass transition temperature. When the wires are cooled, the polymer will relax to its original featureless state shown in Step 5.
Figure 4-7: A shape memory composite can undergo a surface texture modifying heat cycle by expanding and contracting active axial wires. An ‘accordion’ effect may form on the polymer’s surface.

As discussed in Section 3.3, the initial composites have wires embedded diagonally. Unfortunately, this method proves futile in the attempts to create surface texture. The problem with this method involves the wires shearing through the surface of the polymer as they contract. The contraction of the wires is not centrally located throughout the entire polymer and when heated, the softened polymer gives way to the normal components of the wire contraction. To solve this problem, the wires were strung centrally through the polymer by stringing the wire through punched holes in the Viton cords sidewalls. Unfortunately, this method cannot be used with the thinnest polymers due to the impracticality of punching holes through the very slender cords without ruining their structural integrity. Therefore, this method has proven to work best in the middle thickness of 0.094 inches.

The testing plan was to first individually test 0.010”, 0.015”, and 0.020” diameter wires in the 0.094” thick polymer to see their local effect. The local effect tests would be used to gain knowledge into which wire is the optimal choice to use in the embedding of an array of wires used to cause a global surface effect. When tested, the wires had a minimal local effect on the surface texture. The wires visually contract at Dynalloy’s suggested current values but looked as though they were tunneling through the polymer. The wire-polymer composite clearly is not acting as one when the contraction is taking place. The local effect has not given a ripple appearance, but instead creates a small bulge above the length of the wire. The polymer also did
not have the ability to return the small change in texture to a smooth surface. The polymer bulge is pictured in Figure 4-8. Although the bump is slightly perceptible, the texture change can be noticed best when viewing its diffraction of overhead lighting.

Figure 4-8: Although no ripples were created on the polymer’s surface, raised surface textures were evident by viewing a diffraction of light.

4.2.2 ESEM Imaging

After studying multiple composite samples that caused a minimal surface effect, the wire-polymer interface was viewed under an environment scanning electron microscope. Small sections of polymer were placed between a roll on clip and then set into a two step epoxy to cure overnight. The surface was prepared for polishing using a very fine saw blade to cut the epoxy block and contained composite sections to a half inch cylinder. These cross-sections were then polished with up to 1200 grit wetted sandpaper to clear irregularities from the wire-polymer interface caused by the previous sawing step. After the polishing step, the polymer resembles the sample shown in Figure 4-9. The final preparation step involved applying a thin gold paint to give a higher quality picture under magnification.
The microscopic views shed light onto why the wires are not causing the desired surface effect. Figure 4-10 shows a picture of a Flexinol® wire embedded into a Veriflex® cured polymer. This wire has not been through a heat cycle, so there have been no contraction and relaxation stages. Thus, it is expected that the wire would be nicely encased in the polymer as shown. Figure 4-11 shows a zoomed in ESEM image of the small gap (1.11 µm) between wire and polymer.
The wires that have been through a heat cycle displayed a different wire-polymer interface. As Figure 4-12 shows, there is a much larger gap in heat cycled wires compared to wires that have
been through no heat cycles. The heating is evident due to small char marks on the polymer surrounding the lower half of the wire’s circumference. In Figure 4-13, a magnification of the gap displays that the distance between the wire and polymer is nearly 8 times as large. Also, the polymer’s radius is very rough. This is most likely due to the bond between the polymer and wire being torn apart when the wire contracted and relaxed through a few cycles.

In short, the reason for the wires not creating a larger compressive surface effect is due to the wires expanding their polymer surrounding and leaving a void after the first heat cycle. The wires are no longer bonded with the polymer as the torn radius of the polymer implies and are virtually sitting in space. Therefore, any heat cycles from then forward allow the wire to freely tunnel through a polymer’s charred, possibly glassy, interior passageway and there is no frictional pull. After the bond is broken, the heating of the wire does not distribute enough warmth through the increased open space to allow the whole polymer’s temperature to rise above $T_g$.

![Figure 4-12: A Flexinol® wire (light) resting in a void inside the Veriflex® polymer (dark) created after cyclical heating. An axial contraction created a corresponding enlargement of wire diameter that left space between the polymer and wire.](image)
The lack of a bond led to attempts at other wire embedding techniques to cause more friction. Some examples are shown in Figure 4-14 and these all tended to have similar surface modification difficulties as straight Flexinol® wire.
Figure 4-14: Proposed bonding techniques that were attempted to increase the frictional bond between the polymer and alloy.

Hard TRA-BOND 2106T epoxy droplets effectively bond to the Flexinol® wire but dissolve into the corrosive resin during a cure cycle as pictured in Figure 4-15. Attempts to solder on tin droplets are found ineffective due to a weak attachment between the tin solder and the NiTi wires. The most effective means of increased surface ripple creation is to twist wires into a tight helix pattern as pictured in Figure 4-16. Very small local ripples are found above the helix, but the ripple effect is not deemed possible on a global composite scale. The helix pattern does prove to increase the frictional bond and thus the compression on the local polymer.

Figure 4-15: Epoxy droplets dissolved in the cured polymer. This is another example of the polymer’s volatility.
Figure 4-16: Helix wires were able to create a very small local ripple effect on the polymer’s surface when activated.

4.3 Vacuum Pulled Polymer Results

After the axial wire substandard findings, the research quickly changed gears to an alternate mode of surface activation. An idea formed to use a vacuum pull on a convex or flat heated shape memory polymer to a flat or concave position, respectively. The SMP’s surface disturbance would need to be large enough to create changes in drag similar to the accordion effect previously discussed. In this case, the idea would be tested on a larger scale, and if successful, would be modified to create a network of vacuum activated shape morphing areas.

The vacuum pulled polymer experiments exists in three phases. The first phase looks at pulling a convex polymer to a flat state. The second phase looks at pulling a flat polymer to a concave state. The final phase looks at modifying the second phase to include a passive wire mesh within the polymer to create a self-heating vacuum pulled composite.
4.3.1 Convex Polymer

The original proposed idea was to vacuum a convex polymer onto a flat surface and then return it to its convex shape with added heat. This meant that the cured state of the polymer needs to be the convex shape as the cured state is the memorized state of Veriflex®. As mentioned in section 3.1.2, a parabolic mold was machined for curing the memorized convex parabola.

This parabolic mold is not very successful in practice for a few reasons. First, the mold’s cavity is very thin (0.040 in.) and is very susceptible to air bubbles being trapped. These small air bubbles are formed in the mixing of the two part resin. Also, the mold traps bubbles during the twisting method used to seal the mold. These air bubbles are very detrimental in their weakening of the polymer by creating a permeable surface as shown in Figure 4-17. This figure shows a view looking down at the top of the convex surface that is raised approximately 0.010 inches.

![Figure 4-17: A custom designed convex parabolic polymer formed many bubbles in the molding process.](image)

Second, the CRG mold release does not work as well on the Lexan as compared to the glass sheet causing the polymer to affix to the mold surface. To remove the cured polymer from the
mold, the mold is first pried apart with a slender wedge. In the three individual tests, the polymer stuck inside the concave side of the mold. To remove it, an outline was carved around the base of the convex polymer and wedge was slid underneath. The wedging process contorted the polymer as its bond to the Lexan surface was broken. In two of the three polymers created, the bond between the polymer and Lexan was large enough to snap the polymer during the removal process as shown in Figure 4-18. Fortunately, one convex polymer did survive the removal process. Although its structure was weakened by air bubbles, the polymer in Figure 4-14 could be tested by covering the holes with dabs of silicone sealant.

![Broken Polymer](image)

Figure 4-18: Snapping of the polymer during extraction from the mold. This was caused by failure of the mold release to keep the polymer from sticking to the Lexan material.

To begin testing, an orifice plate needed to be designed to act as an interface between a vacuum pump and the convex polymer. Aluminum was machined to create a 3”x 3”x 0.5” plate with a centrally located 7/16” tapped hole. The vacuum pump’s steel hose connection threaded into the hole and the base of parabolic polymer was attached with super glue to the opposing side. Silicone sealant was also applied to the outer ring of the base to prevent any vacuumed air from escaping the base of the polymer.

The one convex polymer that survived underwent a memorization cycle explained by Figure 4-19. The polymer was heated to 75ºC (13ºC above Tg) with a heat gun and pulled with the
vacuum shown by the blue arrow. The heating allowed for the softened polymer’s molecular switching segments to fix the temporary shape. Once the temporary shape was a flattened polymer, the vacuum was turned off. The polymer was allowed to cool in this position until it became a rigid flat plate. Next, it was time to test the memorization capability of the self-molded polymer bump. Heat was again applied using the heat gun, raising the polymer’s temperature to 75ºC. Slowly the polymer began to reform its original parabolic shape in the red arrow’s direction.

Figure 4-19: Vacuum-heat cycle consists of a vacuum pull (blue) a return using heat (red). First, the rigidly bound convex polymer is heated above T_g. Next, a vacuum is pulled through an orifice plate causing the polymer to flatten. Here, the polymer is cooled and the vacuum is turned off. Finally, the memorization of the formed shape occurs when heat is again supplied to the system.

The vacuumed polymer memory cycle was proven with the molded convex polymer. Due to the difficulties in the molding the unique shape and the unattractive air bubble riddled results, the surface modifications were redesigned and the research took another turn for the better.

4.3.2 Concave Polymer

Mentioned earlier in the report, a major influence for this research is to create surface features similar to a golf ball’s dimples. The dimples are used to create a turbulent boundary layer that results in a thinner wake behind the golf ball and less drag as shown in Figure 4-20. Using the knowledge gained from the previous polymer vacuum tests, the simplicity of curing a flat polymer sheet, and the inspiration of a golf ball’s dimple, the next polymer experiment involves pulling a thin flat polymer into a concavity.
Figure 4-20: Golf ball dimples have a significant effect on drag. Golf balls can fly further and faster due to these aerodynamic surface modifications. These modifications provided inspiration for the final phase of the project.

Again, an orifice plate would need to be machined to act as the vacuum pump and polymer interface. An aluminum plate was machined with a centrally located concavity and tapped hole as shown in Figure 4-21. Circles were cut out of previously cured 0.063” polymer sheets (some included Flexinol® wires that did not affect testing) and were attached to the plate with epoxy shown in Figure 4-22.

Figure 4-21: A concave orifice plate was machined using CNC techniques that located a tapped hole at the center of a concave dish. Polymers could be rigidly attached to the top of the aluminum surface.
The flat-to-concave polymer is actuated in similar method as the convex-to-flat polymer. Heat is applied with a heat gun to raise the polymer’s surface temperature to 75°C. Subsequently, a vacuum is pulled and the polymer deforms onto the concave surface. Heat is removed to allow the polymer to become rigid and then the vacuum is turned off. The deformed shape of the polymer is shown in Figure 4-23. The peak change in depth is 0.270 inches. A return of external heating the polymer slowly returns to its original flat state over the period of 3 minutes.
Now that the flat-to-concave idea had been proven, more tests were performed to see results of increased heating and different orifice plate shapes. First, the polymer was heated to 100°C. This heat causes the polymer to be a great deal softer than the 75°C polymer and therefore, it deforms further into the concavity. To make sure that the polymer does not detach from the epoxy, C-Clamps hold down an aluminum frame around the circumference. The results of the vacuum induced deformation are of a much greater magnitude as shown in Figure 4-24. The polymer was soft enough to deform into the tapped hole resulting in a 0.450 inch deformation. Once again, a heating process can return the polymer to a flat state although the increased deformation required a 5 minute process.

![Figure 4-24: Increased heat (90°C) caused increased deformation into the orifice plate. This polymer achieved a 0.450 inch deformation.](image)

A second experiment tested how small the surface deformation radius of could be. Using the original plate designed for the convex-to-flat polymer, the flat polymer was heated to 90°C and pulled into the tapped hole. A less pronounced but smaller radial concavity formed in the polymer as shown in Figure 4-25. This type of concavity may be a better option for a network of polymer bumps due to its good deformation to radius ratio.

![Figure 4-25: Smaller dimples were created by pulling the polymer into a tapped hole. The deformation was 0.065 inches. These surface features may be reproduced and packed tightly similar to a golf ball’s dimple design.](image)
4.3.3 Resistive Heating Network

Although the results of the vacuum actuated polymers are a recognizable achievement, they are still externally heated with a heat gun and could not be used in real-life controllable applications. There is a desire to include a self heating network of passive wire mesh inside the polymer. The embedding of the mesh into the polymer is solved in the curing repetitions discussed in the Section 3.3. Testing various wire mesh prototypes shows that the hottest areas of the mesh occurred near curves in the wire mesh path and that the coolest areas are in the spaces in between the wire mesh paths. So the best method of obtaining a high average surface temperature is to compact wire mesh curves into a small space. The result is the aluminum wire mesh shown in Figure 4-26.

![Wire mesh with compacted curved paths](image)

Figure 4-26: Wire mesh with compacted curved paths was found to heat the polymer to a high average surface temperature.

This wire mesh is not perfect due to a porous air bubble and low heat values in the unmeshed areas, but it represents the best cured wire embedded mesh so it was selected for the experimental tests. The picture in Figure 4-27 shows the temperature gradient on the surface of the polymer when 3.2 V and 5.1 A were applied through the mesh. The figure also shows the circumference of the applied vacuum pull.
The wires are resistively heated causing the polymer to be softened and the vacuum pull is initiated. Although the embedded wire mesh increased the stiffness of the composite, the mesh is still flexible enough to be deformed into the concavity. The current source is first turned off, then the vacuum, setting the polymer into its new concave position as shown in Figure 4-28.
Figure 4-28: The deformation of the polymer was large enough to reach the bottom of concave orifice plate. The areas between the wire mesh plastically strained during the vacuum process and therefore the polymer could not return to the memorized formed shape.

Unfortunately, there are problems in the memorization stage. The current source is again turned on and the polymer is heated above the wire mesh. The composite begins returning towards its cured flat state, but reaches a stopping point where no extra time under heat has any effect. In all, this polymer sample returned to a 2:1 deformation to thickness ratio.

The reason for the polymer not returning to its flat state is due to the lack of an even temperature throughout the polymer. Small stretch marks in the regions of low heat (under 62°C) show that the polymer did not turn on molecular switching segments and is deforming plastically. These plastic strains cannot be overcome in the heating stage and ultimately create tangible ridges on the polymer surface.
5. CONCLUSIONS

Ultimately, the project is considered a success. The original goal was to create a self heating shape memory composite skin capable of surface texture modifications. Although this was not achieved using alternation of SMP and SMA actuation, a passive wire mesh embedded in the polymer provides an alternative method of heat cycling. The shape memory composite responds to a vacuum pull method of actuation and returns a concave SMC close to its original formed flat state. The achievements in small phases of the project ultimately lead to the successful completion of the shape memory composite.

<table>
<thead>
<tr>
<th>Project Phase</th>
<th>Success or Failure</th>
<th>Aspects of Success/Failure</th>
<th>Future Possibilities</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cure a Polymer Sheet (Section 3.1.1; 3.2)</td>
<td>Success</td>
<td>• Sheet of polymer was created using both the 10 hour and 36 hour cure cycles</td>
<td>• Possible desire for thinner polymer sheets</td>
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<td></td>
<td></td>
<td>• Sheets were created in thicknesses of 0.063-0.118 inches (1.6-3.0 mm)</td>
<td>• Currently limited by diameter of Viton cords</td>
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<td></td>
<td></td>
<td>• Polymers became progressively more transparent and had better surface finish</td>
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<td></td>
<td></td>
<td>• Polymer properties were confirmed in tension tests as well as memory cycle testing</td>
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<tr>
<td>Design a Custom Shaped Polymer (Section 3.1.2; 3.2.2)</td>
<td>Failure</td>
<td>• Lexan parabolic mold created using CNC process with 0.040 inch thick cavity</td>
<td>• Ideally, the mold needs to be created from glass or aluminum</td>
</tr>
<tr>
<td></td>
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<td>• No simple method to fill custom mold</td>
<td>• Glass will be hard to shape into desired mold shape</td>
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<td></td>
<td></td>
<td>• Air bubbles present throughout mold filling process</td>
<td>• Aluminum does not offer transparency to see how mold is filling</td>
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<td></td>
<td></td>
<td>• Mold release did not keep polymer from attaching to Lexan surfaces</td>
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<tr>
<td>Embed Wires or Wire Mesh into a Polymer (Section 3.3)</td>
<td>Success</td>
<td>• Flexinol® wires were embedded into the polymer using both the diagonal and punched method</td>
<td>• Possibilities of using other embedded materials</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Wires were strung through the sides of the upright mold</td>
<td>• Not entirely necessary due to current methods' successes</td>
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<td></td>
<td></td>
<td>• Aluminum screen mesh was corrosion resistant to the volatile polymer resin</td>
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<td></td>
<td></td>
<td>• Copper mesh reacted harshly with resin</td>
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<td></td>
<td></td>
<td>• Best approach was to bring mesh leads above resin surface</td>
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Future considerations should be given to three specific areas. The first area concerns of the heating optimization of the wire mesh. As mentioned previously, the vacuumed composite had uneven heating and therefore the deformations are not 100% reversible in areas that did not achieve a temperature above $T_g$. Eliminating the gaps between the wire mesh patterns may be the first step in achieving a good temperature gradient throughout the wires. Also, other mesh materials should be explored to see if their electrical properties are more beneficial than the current choice of aluminum. The aluminum mesh does provide a lot of flexibility to the composite and future choices should take this into account. Also, it is important that the next iteration of wire mesh is corrosion resistant.
The second area for improvement would be to prove that the polymer sheet can be activated to form a network of polymer dimples. The system can be optimized to pack as many dimples as possible into a small area on the polymer similar to a golf ball’s surface. A new method of constraint may need to be developed. This constraint can possibly be shaped like a honeycomb to keep the circumferences of the dimples clamped while allowing the viewing of the vacuum actuation. In addition, a similarly designed orifice plate with multiple vacuum holes may need to be machined. A control system can be setup to manage the DC current supply as well as the vacuum to take the composite through multiple memorization cycles. Finally, steady state aerodynamic tests of the vacuumed dimple network should be used to determine the overall drag coefficient change.

The ultimate goal of the continuation of this project would be to eliminate the need for the bulky vacuum pump mechanical actuator. This can be done by setting the memorized state of Nitinol wires in a parabolic shape. Embedding the wires in the SMP could supply the necessary heating to cycle the polymer through its heat cycle as well as the means of downward actuation required to create the dimple effects. This is an original goal of this project but ultimately due to a steep initial learning curve and time constraints, this course of action was abandoned for the vacuum means of actuation.
6. BIBLIOGRAPHY


