Self Healing Materials

ENMA 490 Capstone Design Course
Fall 2004

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Introduction

Motivation

Microcrack formation due to fatigue inevitably degrades properties and eventually causes catastrophic failure in structural materials. Detection of microcracks is difficult, and often repair is not an option, especially for inaccessible components.

Previous research has demonstrated that monomer-filled microcapsules and solid catalyst can theoretically be embedded in a thermosetting polymeric matrix. As a microcrack propagates, it is attracted to the microcapsule, and the tip of the crack ruptures the shell, releasing monomer into the crack. As the low viscosity monomer flows into the crack via capillary action, it interacts with the dispersed catalyst and quickly polymerizes. The polymerized monomer bonds the crack faces, thus blunting the crack tip and preventing propagation. While the healed material is not fully restored to its original state, it has been shown that healing recovers a large portion of the original properties. By preventing microcrack propagation, self-healing mechanisms lengthen lifetime and improve reliability.

The ENMA 490 capstone design project consisted of the design and fabrication of a polymer composite designed to heal microcracks autonomously. Initial motivation was provided by a NASA solicitation to develop a self-healing wire. The solicitation and existing literature on self-healing composites formed a basis for designing a novel system.
Objectives

New self-healing polymer systems will extend lifetime of components that are inaccessible for routine maintenance. This project will center on the development of a thermosetting polymer with self-healing properties. Specific monomers, catalysts, supplies, and processing equipment will be needed to carry out the synthesis of the proposed system. Initial research has isolated the components that will be necessary for a high probability of success.

Intellectual Merit

Self-healing is an intriguing mechanism inspired by biological systems that includes many complex interdependent variables. By comparing the results obtained to previous research, we can determine if self-healing is a general phenomenon or matrix material specific.

Broader Impact

Once a self-healing composite has been realized, it is anticipated that the electrical insulation fabricated with this self-healing capability will increase the lifetime and reliability of critical components where catastrophic failure is unacceptable. Similar advantages could be utilized to improve such systems as those used in space flight, undersea remote probes, nuclear power plants, biomedical implants, and remote military surveillance. By demonstrating that the self-healing mechanism can be extended to other polymer classes, future work may include extension of this concept to more general polymeric applications.
Materials Selection

All materials selected were chosen based primarily on their material properties, the previous research, and faculty recommendations. Some other factors influencing material selection included fabrication feasibility, safety and ethical issues, cost, commercial availability, and delivery time.

Matrix Material - Polyurethane

The matrix material selected for this project was polyurethane. This material was selected for a variety of different reasons. In previous research, an epoxy matrix material was used [1]. Polyurethane (PU) is a good choice for a matrix material because it has superior toughness and high abrasion resistance. Additionally, the two-part resin system is highly reactive, has a low viscosity, and a low glass transition temperature. These properties combined make it suitable for processing. Dr. Briber has recommended this material because, in addition to the aforementioned properties, PU shapes are very easy to fabricate by sandwiching the polyurethane mixture between two glass sheets that are held together by binder clips. There are two parts to the polyurethane: part A is a di- or poly-isocyanate, and part B is a polyol. When part A and part B are mixed together, the two parts polymerize to form the polyurethane compound. The structures of these two parts can be seen in appendix B. Polyurethane is also relatively inexpensive and commercially available. Polyurethane is also a relatively harmless polymer and was easy to fabricate in a lab provided by Dr. Al-Sheikhly.
Figure 1. Quik Cast casting resin for polyurethane fabrication [4]

**Microcapsule Shell – Urea Formaldehyde**

Microencapsulation is a widely used process to contain liquids in shells. There are several basic methods, most of which involve the formation of an emulsion. In the case of poly(urea-formaldehyde) (UF) shells, microencapsulation is executed using a variation of interfacial encapsulation called *in-situ* polymerization [5].

There are several specifications that the capsule must meet in order to be successful in the self-healing system. For the shell, the elastic modulus must be less than that of the matrix. If the elastic modulus of the shell is larger, the crack will be diverted away from the capsule. This will succeed in strengthening the composite due to the lengthening of crack propagation distance. However, self-healing will be avoided because the crack tip will go around the capsule rather than pierce its shell. Thus, the self-healing mechanism becomes useless.

Urea-formaldehyde (UF) capsules are very widely used in industry and the process is well studied and understood. This made UF an especially attractive encapsulation medium because scientists and engineers often spend years to decades researching the effect of processing parameters on the properties of the microcapsules
like size, permeability, wall thickness, percent yield, shape, and degree of agglomeration. In this case, UF had been studied by a group at the University of Illinois at Urbana-Champaign (UIUC) and specific recipes had been developed and made available with chemical names and quantities as well as a full, relatively detailed procedure. UIUC had already found success with UF so it was chosen because while we would have liked to have been inventive, we did not have the time or expertise to develop our own process.

The UF selection was found to have another benefit. While writing the proposal for funds, we realized our intention to show that the self-healing mechanism is not exclusive to epoxy composites. The Autonomic Healing research group at UIUC has demonstrated successful self-healing in epoxy composites and has suggested that the same mechanism applies to other polymer matrix materials. However, they did not test any other materials. By keeping all else constant, it is possible to see the effect of the matrix on the self-healing efficiency of the system. If too many variables are introduced, one is not able to determine what has caused the differences. Polyurea capsules were chosen for plan B. However, recipes like those found in White’s U.S. Patent No. 6,518,330 for UF were not available for polyurea so that even though materials were ordered just in case, much work would have to be done to concoct a recipe. Thus the selection of UF was a good decision.

**Encapsulated Monomer – Dicyclopentadiene**

Dicyclopentadiene (DCPD) was the monomer selected to be encapsulated because it has many suitable properties for this application. Dicyclopentadiene is a strained cyclic molecule containing two $\pi$ bonds. Previous research has shown that a solution containing DCPD can successfully be encapsulated in urea-formaldehyde microcapsules. The low viscosity of the DCPD solution allows it to easily fill cracks. To
accommodate the need for an optimum viscosity that will be able to balance the polymerization rate with the rate of crack filling via capillary action, the viscosity can be easily altered with fillers.

When reacted with Grubb’s catalyst a solution of DCPD can rapidly undergo a ring opening metathesis polymerization (ROMP). A schematic of this reaction is shown in Figure 1. The living reaction that occurs requires a monomer to catalyst ratio of 1:1000. Once the reaction is underway it consumes no additional catalyst, which is an important consideration in a self-healing scheme where few Grubb’s catalyst molecules may be contacted by a propagating crack.

![Figure 2. Schematic of the reaction of DCPD monomer with Grubb’s catalyst in a living ring opening metathesis polymerization [1]](image)

Dicyclopentadiene is also practical in terms of financial considerations. The monomer solution is available for approximately 50 cents/lb. From an environmental point of view utilizing DCPD can also be seen as a solution. Large quantities of DCPD are generated annually as waste by the petroleum industry.

**Fabrication**

*Microcapsule fabrication*
Background
Microcapsule design is very important to the self-healing effectiveness of the material. The size of the capsule and the thickness of the shell are important design parameters. The smaller the capsules, the better the probability is that the capsules will be ruptured by a crack since they are more spread throughout the matrix. Also, smaller capsules were shown to have a larger virgin fracture toughness than that of specimens with larger microcapsules [3]. However, smaller capsules interact more due to increased surface area and thus tend to agglomerate. Separation becomes more difficult and surface modification is needed to prevent agglomeration that prevents uniform, random dispersion. In addition, the capsules must contain enough monomer to blunt the crack tip when polymerized and interact with catalyst.

Microcapsule diameter is determined by the stirring rate. According to Brown [1], there is a linear relationship between the log of the mean diameter and the log of the agitation rate. In general, the faster the stirring is, the smaller the capsules are. However, if the rate is too high, a high shear situation develops. This causes a build up of capsules on the reaction beaker [1]. The build up is unrecoverable. Also in [1], it was reported that speeds around 550 rpm are needed for 100 μm capsules.

Adhesion of the capsules to the matrix also affects the self-healing success. To increase the bond strength between the shell and the matrix, a silane wash was performed before mixing into the matrix as reported in the relevant patent [5].

Microcapsule Fabrication Process
The microcapsules were made by adapting a recipe found in the paper "In situ poly(urea-formaldehyde) microencapsulation of dicyclopentadiene" by Brown et al. The procedure is listed in the appendix A. Several adjustments to the recipe were necessary in order to carry out the process in a timely fashion and without spending a lot of money on equipment. First a three bladed mechanical stirrer as described in [1]
was not available. Instead, a low-shear, football-shaped magnetic stirring rod was used as suggested by Von Cresce, a graduate student working for Dr. Kofinas. The DCPD was probably the largest obstacle met in the encapsulation process. Since the DCPD provided by Dr. Kofinas’s lab was a low melting solid at room temperature rather than a liquid as expected, it had to be heated above 33°C before using it for encapsulation. However, despite heating it by running it under hot water, it would quickly cool down, clogging the pipettes. Upon adding the DCPD, it quickly solidified in the solution. Thus, rather than keep the solution at 20-24°C as suggested for stabilization, it had to be heated to around 35°C. This became a problem because the hot plate would not heat up and so solid DCPD spheres formed rather than droplets. Before the formaldehyde was added to start the polymerization reaction, the DCPD had to melt, form droplets, and then stabilize as an emulsion. An often-refreshed hot water bath was used to temporarily raise the temperature and used to help maintain the temperature for later steps. However, even once the hot plate was working, it could not supply enough energy to heat the water bath and solution before the DCPD solidified again. Thus it took much longer than expected (about an hour) to climb to a temperature of 35°C. After stabilizing the emulsion and adding the formaldehyde, the solution was heated to the target temperature (55°C). However, the actual heating rate was much slower than that suggested in the paper of 1°C/min [1].

After maintaining the temperature and stirring for four hours, the capsules were cooled to room temperature. Visually, they appeared to have a large size distribution. There were large spheres of DCPD that had solidified without being encapsulated on the surface, microcapsules, and a lot of what seemed to be UF particles that had failed to attach to a DCPD droplet. The yield was not very good and the capsules agglomerated a lot likely due to the large amount of small UF particles. Also, agglomeration on the beaker walls could be due to high speeds causing a high shear environment.
After diluting the mixture with large volumes of deionized water, the excess solvent was removed by vacuum filtration. The vacuum filtration was very tedious because of the large amount of solids formed. After pouring some filtrant on the center of the paper, it was washed many times with water to help rinse off the solvent. The particles were then dried for about 10 minutes and were recovered into a beaker even though they still reeked of DCPD. This process was repeated for hours until all the capsules were cleaned and dried. Then they were spread out equally into three petri dishes to dry in the hood over the weekend.

Results
To determine if the process was successful encapsulating DCPD, sniff tests were originally planned as well as to break open capsules and to observe them using optical microscopy. The sniff test turned out to not be practical because the DCPD odor was present without even disturbing the microcapsules. Even after washing multiple times and filtering, the DCPD odor was still present. Either DCPD residues were still present on the surfaces or the capsule shells were too thin or permeable. It is likely that the delays due to the hot plate, the different type of DCPD, and type of stirring affected the capsules negatively.

Previous work suggests that while small capsules are desired, the stirring rate used was too high, or at least contained too large a shear component. This high rate likely produced large shear stresses, which is evidenced by the large buildup of capsules on the beaker walls. Since those capsules were recovered and were not separated from the rest, this likely ruined the results. The good capsules should have been separated from the agglomerated ones.

Upon more careful research, silane wash is to be done in a 1:20 ratio of silane to hexane. When silane-washed capsules were mixed into the PU, it did not polymerize
and instead formed a “goop.” There were no polymerization problems however when silane was omitted. Also, White used 1 wt% silane in the polymer, Silane X6032 (Styrylamine Cationic reactive group) [5]. Further investigation is needed to determine the role of the silane.

**Backup Plans**

Since the first round of microcapsules did not go smoothly due to the hot plate problems and the higher than expected DCPD melting point, the backup plan was executed using the plan B material, but only for the monomer. Due to previous practice and the UF recipe, UF capsule shells were maintained since the interfacial polymerization process would work for any water insoluble monomer. Because of the versatility of this type of encapsulation, only the monomer would have to be changed, which was the biggest problem in the first place anyway. Brown in [5] suggested using caprolactone monomer as the core so that was chosen as a backup monomer material. The procedure went very smoothly with everyone contributing and the reaction going as planned. However, an emulsion did not form when the caprolactone was added. At first it was thought that maybe it was because the monomer had similar density, viscosity, and refractive index to water so that we could not see an emulsion. However, when the formaldehyde was added, the solution was still clear. At the target temperature there was still no sign of microcapsule formation. Suspecting failure, the solution was left to react while the caprolactone solubility in water was looked-up. The solubility was to be checked before synthesis, but time was very short and the group was in a rush. It was discovered that caprolactone is one of the few monomers that is soluble in water. Apparently this is something that the UIUC group neglected to mention or realize when writing the patent and their other papers. This mistake shows how important it is to read with careful consideration. Just because something is
published doesn’t mean that it is correct. However, despite the failure, the attempted encapsulation was good practice and went more smoothly than the first encapsulation.

**Matrix Mold Fabrication**

The matrix mold was fabricated using two 8.5” x 11” x 3/8” acrylic sheets held together post casting with large binder clips. The bottom acrylic sheet had a 6” x 8” rounded rectangle with a 0.125” divot cut from it in order to create the hole in which the mixture would be poured into. These dimensions were chosen in order to accommodate later tensile testing. In order to do tensile testing, a dog bone cutter must first be used to cut out dog bone shapes from the sample for the actual testing. This dictated the thickness of the resulting sample. The 6” by 8” size was chosen so that many samples could be cut from the subsequent matrix.

*Figure 3. (a) Mold used to create dog bone tensile specimens, (b) Mold used to create Izod specimens*

**Matrix Fabrication**

We bought two types of polyurethane to use as potential matrix materials. One type was supplied by Polytek and the other by TAP Plastics (QuikCast). Both types came with two parts: part A and part B that polymerize when mixed together and allowed to cure. We began by mixing the first type of polyurethane, Polytek 15-3. This
material was difficult to stir for homogeneous mixing, had a pot-life of 15 minutes and had a cure time of 24 hours. Part A was a tan colored viscous liquid that turned greenish when stirred completely. Part B was camel colored and more viscous than part A; it became milky white when stirred completely. The parts were mixed together with a ratio of 1:1. Combined, the mixture was a medium brown-green color. Bubbles formed which could have been due to moisture in the air or materials. We used this sample to test the adhesion of the matrix to Lexan without spray release and the acrylic mold material with the spray release. Acrylic (3/8” thick) was used instead of glass, and Lexan was proposed as an alternative to Teflon due to availability. After 2 days, the samples were removed.

Next, we tested the polyurethane obtained from TAP Plastics, the QuikCast. This material was easy to shake to ensure a homogeneous sample, had a pot life of 5 minutes before polymerization, a demold time of about 15 minutes, and a total cure time of 36 hours. Part A was very viscous and was a yellowish corn syrup color. We chose to use 120 mL, and because the sample could be pipetted we were able to fairly accurately measure this exact amount. Part B was a thinner liquid than Part A, and a darker yellow, more of a buttercup color. Because the parts needed to be mixed equally, 120 mL of Part B was also used. Part B, like Part A, could also be pipetted, allowing for a more accurate measurement.

Upon mixing the two parts, the solution immediately thickened, and the viscosity continued to increase as it was stirred. The sample turned a cloudy white, and stirring continued for about a minute. In order to test the effectiveness of the mold release, half of the mixture was poured into a petri dish without mold release, and the other half poured into a petri dish with mold release. Both samples polymerized within about 5 minutes, at which time a milky white cloud seemed to expand and consume the sample, turning it all into a cloudy white color. It can also be noted that the sample without mold released turned white or polymerized faster than the sample with mold
release. Once polymerization had begun, it only took a matter or minutes for the entire sample to polymerize and become hard. The reaction was exothermic as the sample become much warmer to the touch after the two parts were mixed.

Additionally, another matrix mold was fabricated to cast samples for use in an Izod test. Ten holes measuring 3” by 0.5” were cut from a 0.5” sheet of acrylic. Two sheets of acrylic were used to sandwich the cut acrylic. The sandwiching sheets had three helical holes cut to allow screws to be put in so that the sheets could be separated from the middle layer after casting as PU tends to spill out of the sides of the holes and cause the sheets to stick together.

Figure 4. Polymerization of polyurethane

Matrix Fabrication Results

The fabricated matrix was an overall success after several fabrication attempts. The first batch of polyurethane (PU) matrix was fabricated from Polytek polyurethane parts. The resulting PU was a light brown color that was very porous yet very hard.
The first PU fabricated from the QuikCast system was a creamy white color that showed significantly less pores than the Polytek PU. The QuikCast PU polymerized much faster than the Polytek, as we were able to witness the actual polymerization within about a five minutes time after parts A and B had been mixed. The demold time for the QuikCast PU was also much faster than the Polytek, and the total cast time was about 36 hours.

One of the first drawbacks we found in fabricating the matrix was the onset of bubbles on the matrix surface after pouring the mixed PU into the mold. This problem was solved by sliding the top mold piece across the lower piece to eliminate the amount of air that reached the surface and got caught under the mold.

Figure 5. Molded polyurethane with obvious excess bubbles
Another problem occurred when excess PU leaked out into the sides of the mold and polymerized between the two sheets. This made demolding difficult as the excess PU made it difficult to pull the acrylic sheets apart. We solved this problem by creating another mold with screw holes in it. This allowed for screws to be inserted to push apart the acrylic sheets to remove the molded PU.

Testing and Characterization

Microscopy

Optical characterization was performed using the Olympus BLX microscope connected to a CCD camera and video system with which pictures could be taken. Magnifications of 100, 200, and 500X were used. Microscopy was done on samples of the microcapsules subjected to various amounts of filtration and on the polyurethane that was embedded with silane washed microcapsules. Characteristic particles of each filtration level are shown in figure 6 as well as a characteristic view of the polyurethane. Notice that in part a of figure 6, the first level of filtration shows a particle that is approximately 200 μm in diameter, and the particles shown in part b of figure 6 are from the second level of filtration and have a diameter of approximately 100 μm. Part c of figure 6 shows a particle from the maximally filtrated particles; notice a diameter for this particle is approximately 50 μm. Consequently, it can be seen that when subjected to more filtrations, the characteristic particle size of the representative samples decreases. More work must be done to determine if the decreasing particle size has a direct correlation to the amount of filtration or if these findings are purely coincidental. Part d of figure 6 shows silane washed microcapsules in the polyurethane matrix. The matrix did not polymerize in the presence of the silane, and therefore light can be seen
transmitting through the matrix. There are air bubbles present indicated by black-rimmed spheres. It is difficult to tell if there are microcapsules present in this view. There was extremely little crystallinity present in the sample. It also appears that the shell does not provide complete coverage of the DCPD as seen in figure 6a.

![Figure 6. Characteristic microcapsules from (a) one filtration, (b) two filtrations, (c) multiple filtrations and (d) microcapsules (washed in silane) embedded in polyurethane](image)

Characterization of the microcapsules and polyurethane embedded with silane washed microcapsules was done using the environmental scanning electron microscope (ESEM). The ESEM is used for samples that are not electrically conducting. First, the microcapsules from a single filtration were characterized. Part of the sample was crushed using the tip of a pen to see if the DCPD would be released and if it could be seen. The results are shown in Figure 7. Note the appearance of a spherical particle with a relatively rough surface in part a. In part b, agglomerations of small, smooth, spherical
particles are seen. It is suspected that these particles are monomer released from the crushed particles. However, it is also possible that they are tiny particles of UF since it seems that these spheres form the rough outside layer of the UF shell.

![Figure 7. (a) Characteristic particle from sample subjected to a single filtration, (b) suspected monomer particles](image)

Next, the sample of microcapsules that was filtrated the maximum amount was characterized with the ESEM. The results are shown in figure 8. In part a, a characteristic agglomeration of particles is shown. The section of the micrograph covered in a purple box in part a is shown at a higher magnification in part b. Notice that the particle is much smaller than that shown in figure 7. Also notice that the surface is rougher, and there appear to be small particles similar to those shown in figure 7 (b) in the rough outer surface of the particle.
Figure 8. (a) Characteristic agglomeration of particles found in maximally filtrated sample of microcapsules, particle in box is same as that shown in part (b)

Finally, the ESEM was used to compare the microcapsules of the research team to those provided by the group at University of Illinois. In figure 9, the surface of a particle that had been filtrated multiple times is shown in part a; a particle from the researchers at UIUC is shown in part b. Notice the similarities in the rough outer surface of urea formaldehyde and a smooth inner surface. The particle shown by the UIUC group has opened and one can see the thickness of the shell wall. This was not possible to do for the particle shown in part a.

Figure 9. (a) Surface of microcapsule from research group at University of Maryland, and (b) Surface of a microcapsule from research group at University of Illinois at Urbana-Champaign [1]
Tensile testing was carried out according to ASTM standard D638 using a Sintech 20 universal testing machine as shown in Figure 10(a). Dog-bone shaped tensile specimens were cut out of as-cast plaques using a dog-bone cutter shown in Figure 10(b). The test specimens, one of which is shown in Figure 10(c), were ASTM Type V having an overall length of 2.5 in., a width in the narrow section of 0.125 in. and a thickness of 0.16 in. The crosshead displacement rate was set at 0.05 in/min.
Figure 10. (a) Sintech 20 universal testing machine used for tensile testing, (b) Dog-bone cutter used to make tensile test specimens, (c) ASTM Type V specimen cut from mold
Five (5) test specimens of the as-cast virgin polyurethane were tested under tensile load until failure occurred. Due to the limitations of the computerized data acquisition system, output files of the test data sets could not be generated and therefore digital images of the on-screen display were captured as an alternative. Figure 11 (a) shows the typical displayed output data for one of the tensile tests. Figure 11 (b) is the corresponding load versus elongation curve that was generated by the data acquisition software during the same tensile test. The generation of stress-strain curves for each of the tensile tests could not be accomplished without the output numerical data files.
A summary of the computer generated tensile test data is provided in Table I. The data show the as-cast virgin polyurethane had an average tensile strength of 3154 psi (22 MPa). The manufacturer’s property data sheet for the Quik-Cast polyurethane
from which these samples were molded, reported a tensile strength of 5000 psi (34 MPa). The difference between the manufacturer’s reported tensile strength and the average test value can most probably be attributed to the presence of stress concentrations in the form of molding defects in the as-fabricated test specimens leading to reduced tensile load capacity. The average elastic modulus was found to be approximately 98,000 psi (676 MPa). A strain gage extensometer was not available to measure the elongation (deformation) of the specimens during testing.

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Table 1. Tensile testing results of the virgin polyurethane samples

**Impact Testing**

Izod impact testing was carried out according to ASTM standard D256 on a Testing Machines, Inc. Izod impact tester located at Adell Plastics, Inc. (Baltimore, MD) and is shown in Figure 12(a). As-cast specimens are shown in Figure 12(b). The test specimens had an overall length of 3 in. and a square cross section of approximately 0.5 in. Prior to impact testing, each of the as-cast specimens was notched at Adell Plastics using a milling machine as shown in Figure 12(c).
Five (5) notched Izod specimens of the as-cast virgin polyurethane and one (1) polyurethane sample embedded with 5 wt. % microcapsules were tested. A summary
of the results of this testing is provided in Table II. The data shows the as-cast virgin polyurethane had an Izod impact resistance of 0.56 ft-lbs/in. Each of the Izod test specimens cleanly broke in a brittle fracture mode emanating from the notch. The results of the Izod impact tests are summarized in Table II. Both the as-cast virgin polyurethane and the polyurethane embedded with microcapsules specimens exhibited areas of porosity. The Izod polyurethane embedded with microcapsules had a large visible area of porosity coincident with the milled notch and also exhibit general porosity throughout as shown in Figure 13. This may explain the lower tested impact resistance value of the microcapsule embedded specimen as compared to the virgin polyurethane specimens.

Figure 13. Izod test specimen embedded with microcapsules showing porosity
<table>
<thead>
<tr>
<th>Virgin PU</th>
<th>Units</th>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
<th>Sample 4</th>
<th>Sample 5</th>
<th>Average Values</th>
<th>Std</th>
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<tbody>
<tr>
<td>Thickness</td>
<td>in.</td>
<td>0.45660</td>
<td>0.44385</td>
<td>0.45680</td>
<td>0.44790</td>
<td>0.44330</td>
<td>0.44969</td>
<td>0.00</td>
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<tr>
<td>Izod Value 1</td>
<td></td>
<td>0.280</td>
<td>0.299</td>
<td>0.335</td>
<td>0.258</td>
<td>0.276</td>
<td>0.290</td>
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<tr>
<td>Resultant Izod Average</td>
<td>ft-lbs/in.</td>
<td>0.53</td>
<td>0.59</td>
<td>0.66</td>
<td>0.49</td>
<td>0.54</td>
<td>0.56</td>
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</table>

<table>
<thead>
<tr>
<th>PU w/ microcapsules</th>
<th>Sample 6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness</td>
<td>in.</td>
</tr>
<tr>
<td>Izod Value 1</td>
<td></td>
</tr>
<tr>
<td>Resultant Izod Average</td>
<td>ft-lbs/in.</td>
</tr>
</tbody>
</table>

Table 2. Izod impact test results for samples of polyurethane with and without embedded microcapsules

**Contact Angle**

Contact angle measurements were performed in order to determine the wettability of the polyurethane substrate with the DCPD. The smaller the angle is, the more wetting the liquid is. A small angle is desired in this case so that the monomer will flow into the crack. If the monomer does not spread, then it will contract into itself and inhibit flow. This non-wetting situation will hinder and most likely prevent the self-healing mechanism from occurring.

To do the measurements, DCPD was warmed under hot water to melt. Using a plastic pipette, several drops were placed on the surface of clean polyurethane. With a digital camera (Figure 14), pictures of the drop were taken in order to measure the angle and estimations were made with only the naked eye. The angle was found to be 19-23 degrees using the picture below and a protractor. Heating the DCPD to higher temperatures will likely decrease the angle and increase wetting. To increase wetting further, a surfactant could be added to the DCPD to increase compatibility between the surfaces and thus increase wetting.
Figure 14. Contact angle measurement of DCPD on polyurethane. The angle was measured as 19-23 degrees

**Fourier Transform Infrared Spectroscopy**

The chemical identity of various reagents can be analyzed with Fourier transform infrared spectroscopy (FTIR). Our starting materials and synthesis products were examined using this method. A reference spectrum for DCPD is shown in figure 15(a) [2]. The results of the analysis of the product of our microcapsule synthesis are shown in figure 15 (b). Due to time constraints, analysis of the FTIR results was incomplete.
Future Work

The project research has only begun. The first step is to fabricate more microcapsules using the DCPD that is a low viscosity liquid at room temperature. Once microcapsules of proper size are created, the next step is to integrate the entire system of microcapsules and catalyst into the matrix. Once the process is cemented, the relative amounts of catalyst and microcapsules should be altered to find the best ratio. In addition, the weight percent of microcapsules in the matrix should be varied along with
the capsule diameters and shell thickness. All these variables need to be explored to determine what provides the best self-healing effectiveness. Experimental tests must be used since theory is not developed enough to explain the phenomenon.

Also to increase effectiveness, the silane wash should be further investigated to determine why it prevents polyurethane polymerization. It is likely the silane chosen was not appropriate for this system even though it was for the epoxy system. Adhesion studies between UF and PU should be done using different surface treatments. To increase the speed of recovery and crack filling, surfactants or other additives could be incorporated into the DCPD to increase wetting and spreading.

To analyze the self-healing effectiveness, further failure analysis must be completed. Izod testing of the complete system should be done to determine the fracture toughness of the virgin, and then pre-stressed and healed composite. Failure due to fatigue is being prevented so fatigue testing should also be executed to determine the degree of self-healing success.

Conclusions

The concept of a self-healing is a relatively new area of technological interest with many potential applications to fields ranging from electronics to biomedical interest. The inherently complex interaction of design parameters in a composite system makes it difficult to apply theoretical models to explain macroscopic properties. Nevertheless, the ENMA 490 design team developed a methodology so that qualitative and quantitative data could be obtained and used to establish criteria for successful self-healing materials.

A variety of experimental techniques were used to understand various aspects of materials science and engineering challenges necessary to fabricate a composite material. While the mechanical test specimens did not show an overall increase in tensile strength, it was learned that other testing criteria are necessary to argue
persuasively for the use of self-healing. The observations of microstructure suggest that a general microencapsulation process can be adapted to diverse polymer matrices with careful attention to surface characterization.

The design project was of great utility in preparing the students for encountering real world engineering problems. The critical thinking necessary to prepare a presentation for a specific audience and to justify a budget served to enhance the overall educational experience.

**Acknowledgements**

We would like to thank the following people for their help with our project. For faculty consultations: Dr. Al-Sheikhly, Dr. Briber, and Dr. Kofinas; for the use of labs and laboratory materials: Dr. Al-Sheikhly, Dr. Kofinas, Dr. Martinez-Miranda, Bani Capriano and Dr. Raghavan, and Dr. Lloyd; for assistance in labs: Jung Chul An, Von Wald Cresce, Alia Weaver; for assistance with ESEM: Tim Zhang, for the use of video equipment: Exponent, Inc.
References

Appendix A: Microcapsule Synthesis Procedure;
Urea Formaldehyde Encapsulating DCPD

Chemicals
- 5.0 g urea
- 0.5g resorcinol
- 0.5g ammonium chloride
- 50mL 2.5 wt% solution of EMA
- 10 wt% NaOH solution to raise pH
- 60mL DCPD (dicyclopentadiene), purified by filtration and vacuum distillation
- 1-octanol (1-2 drops)
- 37% formaldehyde (12.67g) solution \( \rightarrow 11.7 \text{ mL} \)
- DI Water

Equipment/Supplies
- Balance to measure mass of solids
- 1000mL beaker
- Graduated cylinder to measure volume of liquids
- pH strips
- Mechanical mixer (3-bladed propeller)
- Hot plate (programmable if possible)
- Temperature controlled water bath
- Coarse fritted filter
- Vacuum filtration supplies

Preparation Steps:

1. Make EMA solution. Solution is 2.5 wt% of EMA in water.
2. Make 10% NaOH solution
3. Determine mixing speed of mixer.
4. Skills—Vacuum Filtration, need another procedure for this
**Procedure**

1. Add 50 ml of 2.5 wt% aqueous solution of EMA copolymer to 200 ml of deionized water in a 1000 ml beaker @ room temperature (20–24°C).

2. Suspended beaker in a temperature-controlled water bath on a programmable hotplate with external temperature

3. Agitate solution with a digital mixer driving a three-bladed, 63.5mm diameter low-shear mixing propeller placed just above the bottom of the beaker.

4. Under agitation, dissolve 5.00 g urea, 0.50 g ammonium chloride and 0.50 g resorcinol in the solution.

5. Raise pH from 2.60 to 3.50 by drop-wise addition of sodium hydroxide (NaOH) solution.

6. Add 1-2 drops of 1-octanol to eliminate surface bubbles.

7. Add slow stream of 60 ml DCPD to form an emulsion.

8. Stabilize for 10 min.

9. Add 12.67 g (11.7mL) of 37 wt% aqueous solution of formaldehyde (1:1.9 molar ratio of formaldehyde to urea)

10. Cover and heat emulsion at a rate of 1 C/min to the target temperature of 55 C.

11. Agitate 4 h and maintain temperature. Then switch off mixer and hotplate. Cool to ambient temperature.

12. Separate under vacuum with a coarse-fritted filter the suspension of microcapsules.

13. Rinse with deionized water. Air dry for 24–48 h. A sieve was used to aid in separation of the microcapsules (USA standard testing sieves, W. S. Tyler).
Appendix B: Material Safety Data Sheets

Chemical structures from Cambridge Software, ChemFind.

### Urea

<table>
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<tr>
<th>Physical description</th>
<th>CH₃N₂O</th>
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<tbody>
<tr>
<td></td>
<td>White crystals or powder, mild aromatic odor</td>
</tr>
<tr>
<td></td>
<td>MW: 60.0554</td>
</tr>
<tr>
<td></td>
<td>Density: 1.335</td>
</tr>
<tr>
<td></td>
<td>MP: 135 °C</td>
</tr>
<tr>
<td></td>
<td>100% Water soluble</td>
</tr>
<tr>
<td>Hazards</td>
<td>May cause irritation, particular on damp skin</td>
</tr>
<tr>
<td>Use/Purpose</td>
<td>Aqueous wall former</td>
</tr>
<tr>
<td>Amount Used</td>
<td>5 g</td>
</tr>
<tr>
<td>Amount Purchased</td>
<td>500 g</td>
</tr>
<tr>
<td>Supplier</td>
<td>JT Baker</td>
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<tr>
<td>Purity/Grade</td>
<td>ACS Reagent</td>
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### Resorcinol / 1,3-benzenediol

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<th>Physical description</th>
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<tr>
<td></td>
<td>MW: 110.11</td>
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<tr>
<td></td>
<td>Density: 1.272</td>
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<tr>
<td></td>
<td>MP: 110-113 °C</td>
</tr>
<tr>
<td></td>
<td>100% water soluble</td>
</tr>
<tr>
<td></td>
<td>White to off-white needle crystals, slight characteristic odor. Becomes pink on contact with air, light or iron. HYGROSCOPIC.</td>
</tr>
<tr>
<td>Hazards</td>
<td>Strong irritant to skin; Inhalation of dust causes irritation to respiratory tract</td>
</tr>
<tr>
<td>Use/Purpose</td>
<td>Make formaldehyde resin</td>
</tr>
<tr>
<td>Amount Used</td>
<td>0.5 g</td>
</tr>
<tr>
<td>Amount Purchased</td>
<td>100 g</td>
</tr>
<tr>
<td>Cost</td>
<td>$26</td>
</tr>
<tr>
<td>Purity/Grade</td>
<td>ACS 99+% Crystal (Alfa Aesar)</td>
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<tr>
<td>Storage</td>
<td>Keep in a tightly closed container. Store in a cool, dry, ventilated area away from sources of heat or ignition.</td>
</tr>
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</table>
Formaldehyde

| Physical descrip. | CH₂O  
| MW: 30.0262  
| BP: 96 C  
| FP: 60 C  
| Density: 1.083  
| pH 2.8  
| Very soluble, colorless liquid, pungent odor detectable at 1 ppm |

| Hazards | Corrosive--Causes burns. Very toxic by inhalation, ingestion and through skin absorption. Readily absorbed through skin. Probable human carcinogen. Lachrymator at levels from less than 20 ppm upwards. Very destructive of mucous membranes and upper respiratory tract, eyes and skin. |

| Use/Purpose | Organic wall former, for resin |
| Amount Used | 11.7 mL |
| Amount Purchased | 500 mL |
| Purity/Grade | Fisher 37% ACS |

DCPD (Dicyclopentadiene)

| Physical descrip. | C₁₀H₁₂  
| MW:132.2048  
| Density: 0.986  
| MP: 33 C  
| BP: 170 C  
| Insoluble organic, colorless to pale yellow, mild camphor like odor |

| Hazards | May cause skin, respiratory irritation. |

| Use/Purpose | Core/monomer |
| Amount Used | 60 mL |
| Amount Purchased | Use Dr. Kofinas’s |
| Cost | free |
| Purity/Grade | BHT Stabilized, from Aldrich |
## EMA Copolymer
(Ethylene Maleic Anhydride)

| Physical description | insoluble  
|                       | Density: 0.92  
|                       | White powder, characteristic waxy odor  
| Hazards               | Maybe mild skin irritant. Dust may be irritating  
| Use/Purpose           | Additive, emulsifier  
| Amount Used           | 1.25 g  
| Amount Purchased      | 16 oz  
| Cost                  | Free sample  
| Supplier              | Zeeland Chemicals  

## Sodium Hydroxide

| Physical description | HNaO  
|                      | MW: 39.99707  
|                      | White, deliquescent solid water soluble, exothermic dissolution  
|                      | Density = 2.13  
| Hazards              | Corrosive, hygroscopic  
| Use/Purpose          | Raise pH (base)  
| Amount Used          | Made 10 wt% solution  
| Amount Purchased     | 500 g  
| Purity/Grade         | Pellets, ACS reagent from JT Baker  

Ammonium Chloride

| Physical descript. | NH4Cl  
|                   | MW: 53.49  
|                   | Density: 1.53  
|                   | MP: 338 °C  
|                   | White, odorless powder, pH ~5, HYGROSCOPIC.  
| Hazards           | Skin and respiratory irritation.  
| Use/Purpose       | hardener for formaldehyde-based adhesives  
| Amount Used       | 0.5 g  
| Amount Purchased  | 500 g  
| Purity/Grade      | Fisher USP/FCC  

1-octanol

| Formula          | C₈H₁₈O  
| Hazards           | Mild skin irritant, respiratory tract irritation. Flash point: 81°C (178°F)—Combustible Liquid and Vapor!  
| Use/Purpose       | To eliminate surface bubbles  
| Amount Used       | 1-2 drops  
| Amount Purchased  | 1 mL  
| Purity/Grade      | 99+%, Acros Organics  

Appendix C: Committee Organization

6 Committees- each with a chair and vice chair

- **Organization**: Co-chairs; Erin and Melissa
- **Research**: Chair- Paul; Vice chair- Jo
- **Fabrication**: Chair- Erin; Vice chair- S.Paul
- **Testing and Characterization**: Chair- Melissa; Vice chair- Paul
- **Simulation/Calculation/Visual**: Chair- Jo; Vice chair- Erin
- **Paper and Presentation**: Chair- S.Paul; Vice chair- Melissa
Appendix D: Timeline

Week by Week Project Schedule

- Week 1: Research - Choose Topic
- Week 2: Fabrication - In Lab
- Week 3: Preliminary Testing
- Week 4: Preliminary Characterization
- Week 5: Calculations
- Week 6: Presentation/Paper
- Week 7: Testing/Characterization
- Week 8: Research - Materials Used
- Week 9: Research - Crack Propagation
- Week 10: Research - Optimization
- Week 11: Fabrication - Research
- Week 12: Research - Choose Topic
## Appendix E: Budget

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<tr>
<td></td>
<td></td>
<td>Tensile</td>
<td></td>
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<td></td>
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**Total accounted for**: 834.87
Appendix F: Recommendations

These are our comments on the class and suggestions for the future of ENMA490:

- Have time in class without the professor present, especially at the beginning of the class. We have no other time to meet as a group and need to update each other without pressure and comments that interrupt our mindsets.
- Before spending a lot of time at the library or on the Internet, talk to professors once you have an idea and some constraints. They are a fantastic resource and save you a lot of time and pain/stress.
- Pick a topic as soon as possible and have several backup ideas. Once you have looked deeper into the topic, as long as it seems feasible, get started immediately.
- Keep on task. The end of the semester comes faster than you think. Set goals for each week, each class, and each quarter, and always keep the end in mind.
- More emphasis on learning teamwork, project design, the process of research and less on being innovative. There was a large amount of pressure to do something new and high tech, which makes it very easy to get over your heads. There is too much focus, at least in our class there was, on success of the project, on invention, and not enough on learning the process.
- An intermediate review with faculty—will introduce them to what you are doing and expose any "showstoppers." Faculty have a wide variety of expertise and they can offer suggestions in their field. Also, they will be able to see where you are in the beginning and how you end up, see the amount of progress.
- Compare and contrast the design process in industry and in academia. Discuss how this context places constraints on the design process.
- Don't schedule macro, kinetics and senior design all in the same semester. Breaking it up would be helpful.
- For general curriculum: It is hard and for large classes, not feasible to do fabrication. However, that is what we learn. We have no background to do simulations; we have almost no experience with software other than PowerPoint and Excel. This puts us in a very difficult situation.