

Mechanical Testing System Coupled with an Environmental Chamber for Hydrogels

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ABSTRACT

Hydrogels have many biomedical applications. Different applications require varying mechanical properties. The mechanical properties of hydrogels are dependent on their composition and surrounding temperature and pH. There is limited published work to describe these properties and conditions. Our team aims to identify the dynamic stress-strain relationship, creep, and ultimate stress and strain under varying temperatures (4 to 40 °C) and pH levels (2 to 10). Before the mechanical properties of hydrogels can be tested, the individual hydrogel samples must be made. This requires developing a stencil procedure to make the hydrogel samples and building an environmental chamber for the mechanical testing system used. So far this semester, our team has written a stencil procedure and begun investigating the requirements of the materials and maximum dimensions for the environmental chamber.

INTRODUCTION

Hydrogels are water-swollen, cross-linked polymeric structures. They can absorb water or biological fluids and have numerous biomedical applications: drug delivery, tissue engineering scaffolds, and wound healing. Biomedical products may include skin adhesive hydrogels for wound/burn care and electrodes, and anti-thrombogenic, biocompatible, lubricious medical device coatings [1]. Although hydrogels have been extensively used in biomedical research, there is limited published work that presents findings on the physical changes of hydrogels to a specific environmental condition and how these changes affect the specific hydrogel biofunctions and applications [2].

An understanding of hydrogels' mechanical characteristics is essential. The mechanical property of biomedical gels allows researchers to evaluate the material intended for a particular application [3]. The ultimate goal of our design is to elucidate the dynamic properties of hydrogels in response to changes in their environment, such as pH and temperature.

DESIGN OBJECTIVES

Our client, Prof. Weiyuan John Kao, is a professor in the School of Pharmacy and also has an appointment in the Biomedical Engineering Department at the University of Wisconsin-Madison. Prof. Kao would like our team to identify the following mechanical properties of hydrogels: the dynamic stress-strain relationship, creep, and ultimate stress and strain under environmental conditions. These properties will be observed under varying temperature (4 to 40 °C) and pH levels (2 to 10), which mimic biological conditions. The structure and environment (pH, temperature, and blood compounds) regulate hydrogels' degradation [3]. These environmental factors are critical in affecting the mechanical and chemical property of biomedical materials. Research has shown that hydrogels that undergo changes in pH levels and temperature often degrade, exhibit swelling changes, and lose their elasticity [4]. These changes are also dependent on the composition of the hydrogels. Therefore, the composition of the hydrogels will be varied during the determination of the mechanical properties to observe these effects.

Before the mechanical properties of hydrogels can be tested, the individual hydrogel samples must be made. In order to complete this task, a procedure must be developed to prepare a stencil from which to make the hydrogel samples. The testing equipment to be used must also be established, including an environmental chamber, which must be designed in order to vary the pH and temperature.

Stencil Procedure

Our client has specified that the hydrogel samples must be a “dog-bone” shape as required for testing by the American Society of Testing and Materials (ASTM). The approximate dimensions for the samples are 280 μm thickness, 11 mm gauge length, and 2 mm neck width. The stencil procedure is desired to be a fast, exact method that ensures uniform

thickness of the stencil and produces as many hydrogel samples as possible at one time. This procedure should also be approved by the ASTM. Having an approved and uniform stencil will prevent variance during the sample preparation, which could have a significant effect in our mechanical properties data. The product design specification for the stencil procedure is attached in Appendix A.

Environmental Chamber

Our client has specified that the environmental chamber must maintain the temperature and pH of the buffer solution used during the mechanical tests; not interfere with the stress, strain, and creep tests; and be compatible with the Instron 1000 and the Instron 5548 mechanical testing systems shown in Figure 1. The Instron 1000 is located in 1313 Engineering Hall and the Instron 5548 is in Prof. Wendy Crone’s lab in the Engineering Research Building. The chamber must maintain the temperature of a solution in the range of 4 to 40 °C and the pH in the range of 2 to 10. The product design specification for the environmental chamber is attached in Appendix B.



(www.instron.com)

Figure 1. The environmental chamber is to be designed for use with the Instron 1000 (shown on the left) and the Instron 5548 (shown on the right) mechanical testing systems.

Therefore, our objective for this semester is to establish a procedure to make stencils, approved by the ASTM, and to design and build an environmental chamber to be used with a mechanical testing system in order to test the mechanical properties of hydrogels.

CURRENT PROGRESS

Stencil Procedure

We have developed an ASTM approved stencil procedure. The stencils are made of Polydimethylsiloxane (PDMS). PDMS is used for several reasons. It is inexpensive, permeable to gases for biomedical applications, transparent, and bonds easily [5]. PDMS is prepared in the laboratory by mixing a PDMS prepolymer and curing agent (SILYARD 184 Silicone Elastomer Kit, Dow Corning, Midland, MI) together and degassing for approximately one hour. To make the stencil, the PDMS mixture is poured over the EPON Master, a silicon wafer, and sandwiched inbetween aluminum disks by placing steel weights on top as demonstrated in Figure 2. The EPON Master, shown in Figure 3, serves as a cookie-cutter to cut out the stencil from the PDMS layer. The transparency is placed on top of the PDMS layer to provide an easy, clean way to remove the top weights. The pyrex disk is placed on top of the transparency to provide a flat, level surface for the weights. The PDMS sandwich is cured for three hours at 80 °C. After curing, the PDMS stencil can be peeled off from inbetween the EPON Master and transparency. Figure 4 shows the final stencil product. The complete stencil procedure is attached in Appendix C.

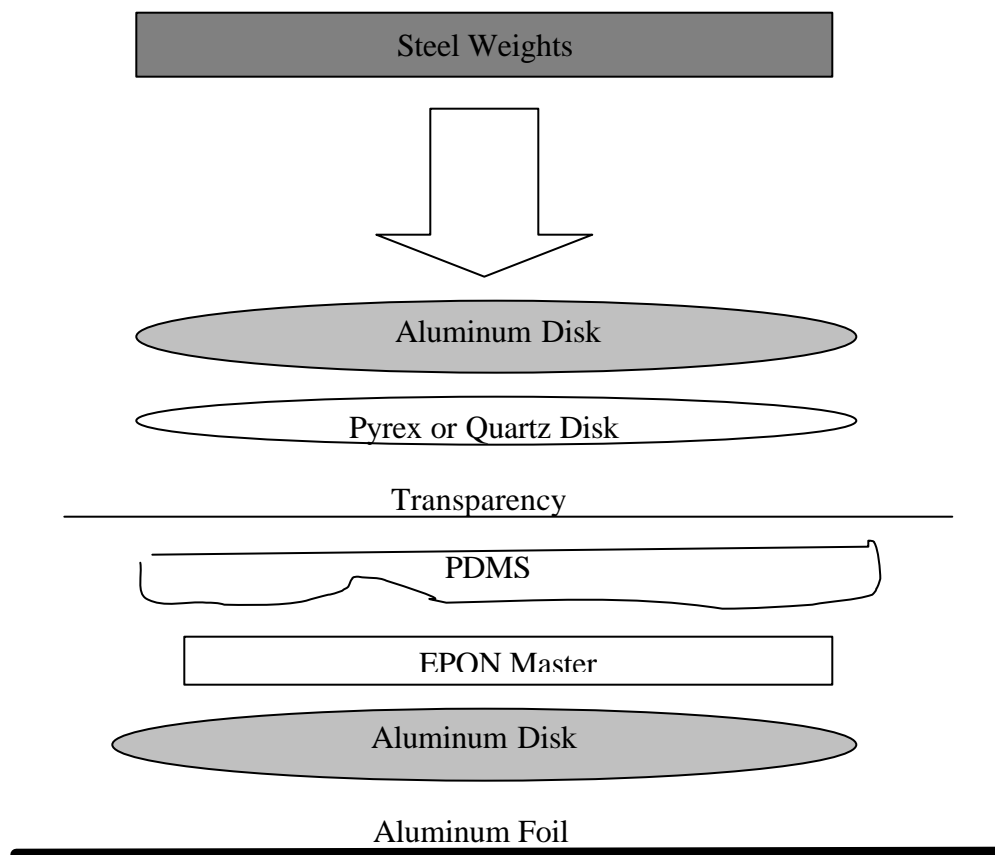


Figure 2. A graphical representation of the PDMS tension stencil procedure assembly.

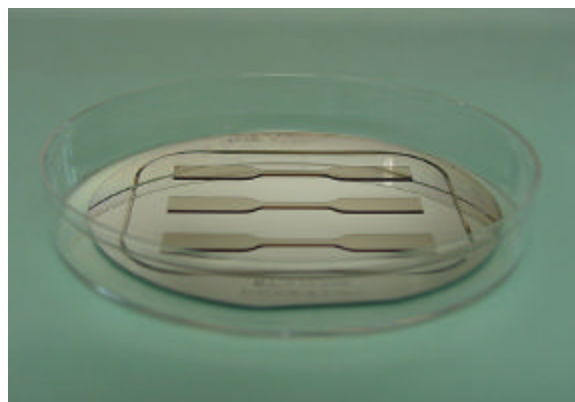


Figure 3. A picture of the EPON Master, which serves as the cookie-cutter for the stencil.

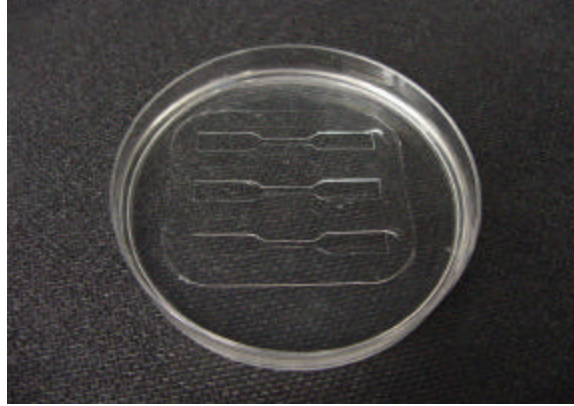


Figure 4. A picture of the PDMS stencil.

Once the stencils are made, they are stored in a petri dish filled with PDMS, which preserves them for up to one week. The procedure for making this base is attached in Appendix D. The entire stencil and base protocol has been tested twice with satisfying results. A similar ASTM, PDMS stencil procedure has been used by Prof. Crone's, on which our procedure is based.

Our stencil procedure has both advantages and disadvantages. It is simple and repeatable, but allows only a limited number of hydrogel samples and is relatively slow. One stencil makes three hydrogel samples and takes four to five hours to make. However, three stencils may be made at once.

Environmental Chamber

We have researched existing environmental chambers: one in Prof. Crone's lab for hydrogel testing and one at the Mechanical Engineering Department at Carnegie Mellon University. The maximum size of the chamber has been determined from measuring the dimensions of the Instron 1000 and 5548 models. The maximum length and width are 20 cm, while the maximum height is approximately 60 cm. We have also brainstormed requirements for the materials from which the environmental chamber will be made. The materials used should

be durable, thermally insulating, transparent, easy to manufacture, resistant to degradation with changes in pH and temperature, and inexpensive.

Simple temperature tests have been performed to determine the decrease in temperature of water in different materials over a three minute period, the approximate time required for one mechanical test of a hydrogel sample. Our client hypothesized that it will be unnecessary to monitor the temperature in the environmental chamber, assuming the temperature drop over three minutes will be negligible. We performed these initial temperature tests to confirm this assumption. We tested the temperature drop of water in a 250 mL pyrex beaker and in a polyethylene container of the same size. The temperature drop over three minutes was two degrees Celcius in the pyrex beaker and zero degrees Celcius in the polyethylene container. These preliminary results indicate that it is reasonable to assume the temperature drop over three minutes is negligible as long as an insulating material is chosen. However, these results must still be confirmed for larger volumes closer to the actual volume of the environmental chamber.

FUTURE WORK

During the second-half of the semester, we will first develop a preliminary design sketch of the environmental chamber. Then, after our client's approval of the design, we will begin researching desirable materials from which we will construct the environmental chamber. Finally, we aim to build and test the environmental chamber. Also, we will implement necessary changes to the stencil procedure after receiving feedback from researchers in Prof. Kao's lab, who will use the stencil procedure.

REFERENCES

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The Product Design Specification of the Mechanical Testing System Coupled with an Environmental Chamber for Hydrogels: Procedure for Making Polydimethyl Siloxane (PDMS) Stencils

Gabriel Martinez-Diaz, Darcee Nelson, Christy Palmer
2/11/02

Function: To establish a procedure to make tension stencils, approved by the American Society for Testing Materials (ASTM).

Client Requirements:

- Fast, exact procedure to make ASTM approved PDMS stencils
- Method to ensure uniform thickness of stencil

Design Requirements:

1. Physical and Operational Characteristics

- Performance Requirements:* The stencil made should be disposable. The procedure should allow multiple stencils to be made at one time.
- Safety:* Be careful when using the hot plate because it is very hot. Although, the materials used are nontoxic, gloves should be worn.
- Accuracy and Reliability:* The mixture of silicone elastomer and curing agent must be homogenous and properly degassed.
- Life in Service:* The stencil is to be used only once and then thrown out. It should also be used within one week after it is made.
- Shelf Life:* The stencil should be stored in the petri dish in which it is made.
- Operating Environment:* The procedure should be carried out at room temperature and normal humidity.
- Ergonomics:* N/A
- Size:* The stencil should be 280 μm thick, have a gauge length of 11 mm, and a neck width of 2 mm.
- Weight:* 10 parts by weight of silicone elastomer should be used to 1 part by weight of curing agent. To make one stencil, use 50 g of silicone elastomer and 5 g of curing agent.

j. *Materials:* The following materials should be used: Sylgard Brand 184 silicone elastomer, Sylgard Brand 184 curing agent, pyrex plates (3 mm thick, 8.5 cm diameter), EPON Master (~280 μm thick, 11 mm gauge length, 2 mm neck width), polystyrene petri dishes (100 mm diameter, 15 mm height), weights – 1 Al plates (3.5 in. diameter, ~102 g) and 3 Al plates (3 in. diameter, ~80 g), tweezers, presentation transparency.

k. *Aesthetics, Appearance, and Finish:* N/A

2. Production Characteristics

a. *Quantity:* One procedure is needed. At least 10 stencils are needed for client.

b. *Target Product Cost:* ?

3. Miscellaneous

a. *Standards and Specifications:* ASTM approval is required.

b. *Customer:* N/A

c. *Patient-Related Concerns:* N/A

d. *Competition:* Prof. Beebe and Prof. Crone have similar procedures.

The Product Design Specification of the Mechanical Testing System Coupled with an Environmental Chamber for Hydrogels: Environmental Chamber

Gabriel Martinez-Diaz, Darcee Nelson, Christy Palmer
3/4/02

Function: To provide a pH and temperature controlled environment for the mechanical testing of hydrogels.

Client Requirements:

- Maintain the pH of a solution in the range of 2 – 10
- Maintain the temperature of a solution in the range of 4 – 44 degrees Celsius
- No interference with the dynamic stress-strain relationship, creep, and ultimate stress and strain tests.
- Compatible with the Instron 5548 and the Instron 1000

Design Requirements:

1. Physical and Operational Characteristics

- l. *Performance Requirements:* The environmental chamber should be able to be used as much as necessary (i.e. consecutive tests).
- m. *Safety:* The chamber should be securely sealed to prevent potentially acidic, basic, very hot, and/or very cold solutions from escaping and causing injury to both the user and any surrounding lab equipment. Gloves should be worn when handling the solutions to be used in the chamber. Oven mitts should be worn if the solution to be used is very hot.
- n. *Accuracy and Reliability:* The chamber should not interfere with the mechanical testing of the hydrogels.
- o. *Life in Service:* The chamber should maintain the temperature and pH of a solution over a time period of 2 minutes, the length of each individual mechanical test.
- p. *Shelf Life:* The chamber should be stored clean and dry in a cool, dry place.
- q. *Operating Environment:* The chamber is to be used at room temperature, atmospheric pressure, and normal humidity. It will also be exposed to solutions with temperature ranging from 4 – 40 degrees Celsius and pH ranging from 2 – 10.
- r. *Ergonomics:* The chamber should be easy to transport.

- s. *Size:* See the Instron 1000 and the Instron 5548 dimensions (to be attached later). The chamber should fit with both of these machines.
- t. *Weight:* The chamber should be light enough to be lifted by one person.
- u. *Materials:* The materials of the chamber should be durable, transparent, easy to manufacture, affordable, insulating, and able to withstand changes in temperature from 4 – 44 degrees Celsius and changes in pH from 2 – 10.
- v. *Aesthetics, Appearance, and Finish:* The chamber should have a transparent shell so that the user can see the hydrogel sample inside. It should also have no sharp edges or extrusions.

2. Production Characteristics

- c. *Quantity:* One environmental chamber is needed.
- d. *Target Product Cost:* ? at this time.

3. Miscellaneous

- e. *Standards and Specifications:* None.
- f. *Customer:* See client requirements.
- g. *Patient-Related Concerns:* N/A
- h. *Competition:* Prof. Crone has an environmental chamber setup in her lab for similar purposes.

Procedure for Making Polydimethyl siloxane (PDMS) Stencils of Tension Samples

1. Put on gloves because the elastomer is very sticky.
2. Measure out (by weight) 10 parts of elastomer (large container) to 1 part hardener (small bottle) using separate clean weighing dishes.
 - a. To make 1 PDMS Stencil, it is recommended to use 10 grams of elastomer and 1 gram of hardener.
 - b. To make 1 PDMS base, in which the stencil will be placed, it's recommended to use 50 grams of elastomer and 5 grams of hardener.
3. Mix the elastomer and the hardener thoroughly with the tines of a plastic fork for approximately 5 minutes.
 - a. If it is not mixed well, the PDMS will not cure properly.
4. Pour the mixture into a jar and degas it under vacuum for approximately 1 hour.
 - a. Make sure all bubbles are gone.
 - b. For larger volumes to make more than 1 stencil and 1 base, it may longer than 1 hour.
5. Place EPON Master on top of aluminum disk, which is placed on aluminum foil.
6. Pour just enough degassed mixture over EPON Master to cover about 2/3 of it.
 - a. Make sure you pour the mixture slowly starting at the center of the EPON Master; this will prevent forming bubbles.
7. Hold weight and EPON master in hand and gently tip it so that the PDMS mixture moves to cover the entire EPON Master.
8. Place a piece of copy machine transparency over the PDMS.
 - a. Make sure transparency has a diameter slightly larger than the EPON master
9. Starting from the center of the master, use a small cylinder (pen or marker) to roll out any bubbles that may have formed under the transparency.
 - a. Roll from the center in one direction, and then starting from the center again, roll in the opposite direction.
10. Place pyrex disk on top of transparency. Place aluminum disk on top of the pyrex disk. Place 2 steel weights on top of the aluminum disk to push out any excess PDMS.
 - a. Hold weights in place as PDMS mixture is pushed out from underneath to prevent the transparency from sliding. Once the setup settles and no longer slides, then may let go.
11. Bake in an oven for 3 hours at 80 °C.

12. When done baking, let cool to room temperature. Then separate components.
 - a. Throw away transparency and aluminum foil.
 - b. Clean off any PDMS residues on EPON Master, pyrex disk, and weights with methanol.

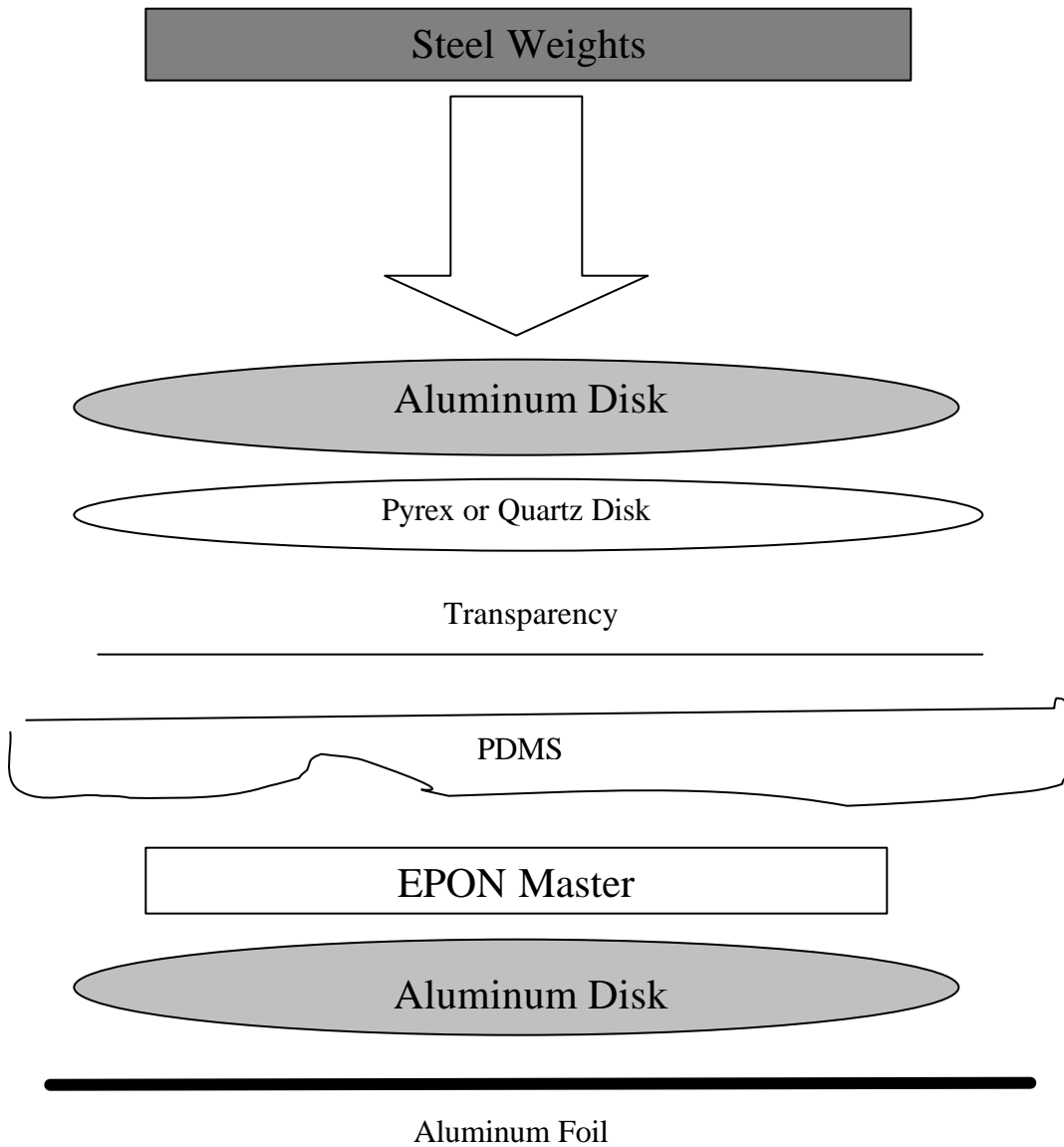


Figure 1. A graphical representation of the PDMS tension stencil procedure assembly.

Base for Tension Samples And Cover Slip for Tension Samples

1. Put on gloves because the elastomer is sticky.
2. Measure out (by weight) 10 parts of elastomer (large container) to 1 part hardener (small bottle) using separate clean weighing dishes.
 - c. To make 1 PDMS Stencil, it's recommended to use 10 grams of elastomer and 1 gram of hardener.
 - d. To make 1 PDMS base, in which the stencil will be placed, it's recommended to use 50 grams of elastomer and 5 grams of hardener.
3. Mix the elastomer and the hardener thoroughly with the tines of a plastic fork for approximately 5 minutes.
 - a. If it is not mixed well, the PDMS will not cure properly.
4. Pour the mixture into a jar and degas it under vacuum for approximately 1 hour.
 - a. Make sure all bubbles are gone.
 - b. For larger volumes to make more than 1 stencil and 1 base, it may longer than 1 hour.
5. Pour degassed mixture into the bottom of the Petri Dish.
 - a. Make sure you pour the mixture slowly starting at the center of the Petri Dish; this will prevent forming bubbles.
 - b. Fill the Petri Dish up to half of its volume.
6. To make the Cover slip, use the top part of the Petri Dish.
 - a. Pour a thin layer of the PDMS solution. It should not reach the border of the Petri Dish. Hold dish in hand and gently tip it so that the layer spreads out towards the edges of the dish.
7. Bake in an oven for 3 hours at 80 °C.