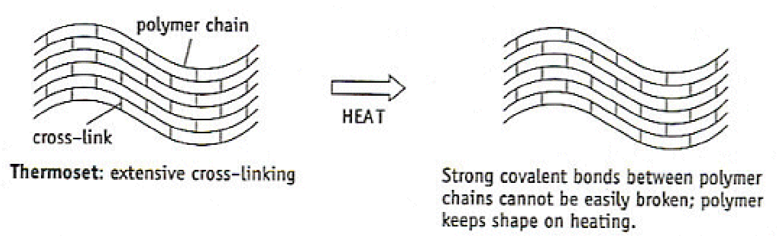
Name: ______________________________________ Class period: ________

<u>Making Shape Memory Monsters: They Come Back!</u>

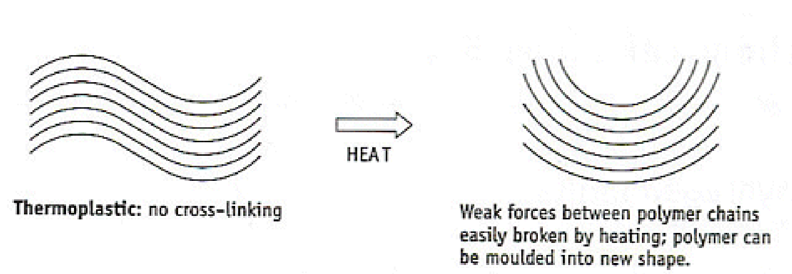
Shape memory materials can be programmed to have a specific shape (such as a pumpkin, ghost, or skull), which they will return to under the right conditions (such as a particular temperature). As a real-world example, imagine having a tire that will repair itself at the right temperature – that's something NASA scientists and engineers are working on right now for the Mars Rover[1]. Other current scientific research is being done on **shape memory materials** made from polymers.

Recall that **polymers** are long chain-like molecules. These polymer chains can become **cross-linked**, a bit like a tangle of necklaces that have formed several knots. Each crosslink connects the strand to other strands, causing the tangled polymer chains to move together when a force is applied. The higher the amount of crosslinking, the tougher and more resistant to stretching that a material becomes.

Thermoset polymer materials have very strong crosslinks that are formed in the chemical reaction when they are first molded. This crosslinking makes thermosets strong, durable materials that can withstand high temperatures (400°F+) without melting, but also makes them difficult to recycle. [2] A silicone baking mold is an example of a thermoset polymer.



Thermoplastic polymer materials have weaker (or no) crosslinking that allows them to be re-molded again and again when heated. The lower intermolecular forces holding them together can be overcome by heating, allowing them to be easily recycled. Plastic bottles are made of thermoplastic polymers.



In this lab, you will be making a **composite** material that has **shape memory** properties. Composite materials take advantage of the different physical and chemical properties of their components. In order for a material to have shape memory, the first component must develop an elastic, thermoset network. In this lab, the thermoset component is the <u>silicone</u> rubber. The thermoset material provides the <u>permanent shape</u>, so that the original molded shape isn't permanently destroyed after applying an external stimulus, such as heat or force.

The second component of the composite material must have a crystalline structure that can be melted and re-formed, restricting the material from recovering until desired. In this lab, the stearic acid will act as the crystalline material.

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[1] "Metal Tires for Mars: 'Shape Memory' Could Help Rovers Roll" https://www.space.com/39305-metal-tires-for-mars-rovers.html
[2] Polymer drawings from "Polymers" http://www.4college.co.uk/as/poly/polymers.php

Name: ______________________________________ Class period: ________

Pre-Lab Questions:

1. What does it mean for a material to have shape memory?

2. How does cross-linking affect the properties of a polymer?

3. What are some of the differences between **thermoset** and **thermoplastic** materials?

4. What is a composite material?

5. What are some benefits of using a composite material?

Lab Part 1 Materials:

- Safety glasses or goggles
- Safety gloves (non-latex)
- Lab scale
- Stearic acid – finely ground in a coffee grinder designated for this purpose (powder-like consistency works best)
- MoldStar20T – Smooth On silicone rubber
- 4 small cups (2 or 3 oz. size works best)
- Metal spatula
- Paper towels
- 3 small plastic molds (any shape will work, but cannot be made of silicone)

Making the Shape Memory Monsters - Procedure

1. First obtain safety glasses or goggles and safety gloves.

2. Get 4 small cups and using a marker or pen, label them separately: A, B, stearic acid, composite.

3. Measure out between 4.5 to 5.0 grams of Part A of the MoldStar20T in the cup labeled A.

4. Measure out between 4.5 to 5.0 grams of Part B of the MoldStar20T in the cup labeled B. MAKE SURE YOUR SPATULA IS CLEAN, OR USE A DIFFERENT UTENSIL IN EACH CONTAINER TO PREVENT CROSS-CONTAMINATION!!!!!

5. Measure out between 0.8 to 1.0 grams of finely ground stearic acid in the cup labeled stearic acid.

6. Stir each cup separately first, wiping the spatula on a paper towel in between to avoid starting the reaction early.

7. Pour cup B into cup A, and mix until thoroughly distributed. Once you pour in part B, you have ~4 minutes to finish mixing and mold your materials before it starts to set.

Name: ___________________________________ Class period: ________

8. Carefully pour a little over half of your A/B solution into the cup labeled "composite." Then add the stearic acid to the composite cup. Add 1 drop of food coloring. Mix thoroughly. Record the color of food dye here: ______________

9. Carefully pour the stearic acid composite into the first 2 plastic molds. Pour into the center of the mold, and allow the material to spread out on its own to fill the edges.

10. Then add 1 drop of a different color of food coloring into your remaining solution from the A/B cup. Quickly stir until distributed, and then pour into the third mold. This will be your control sample. Record the color of food dye here: ______________

11. Use a paper towel to clean the spatula. Throw away the cups in the trash, and then wipe down your counter.

12. Let the samples setup overnight, before continuing onto lab part 2. (The stearic acid will slow the reaction rate, so it will take longer than stated on the box for the MoldStar20T to finish reacting.)

Post-Lab Questions – Part 1

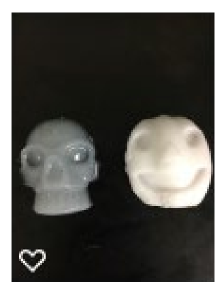
1. What kind of mixture (homogeneous or heterogeneous) was initially made when the stearic acid was mixed with parts A and B? How do you know?

2. What kind of mixture (homogeneous or heterogeneous) was the combination of A and B initially?

3. Why did your group only have around 4 minutes to complete the final steps of the lab? What is happening when the material starts to "set"?

Lab – Part 2 – Fighting the Shape Memory Monsters

Lab Part 2 Materials:



- Safety glasses or goggles
- Safety gloves
- 2 large beakers (400 mL or larger)
- Hot plate
- Thermometer
- Metal spatula
- 3 small binder clips
- Timer

Procedure – Lab Part 2

1. Put on safety glasses or goggles, and safety gloves.

2. Fill both of the large beakers with water to the top line.

Name: ____________________________________ Class period: ________

3. Place one of the beakers on the hot plate, and turn on high. Put in a thermometer to observe the temperature. Heat until ~85°C. You don't want the water to boil, but it needs to stay above the melting point of stearic acid, which is ~70 °C. Once the water is hot, turn the temperature down, but leave the hot plate on until testing is complete. Monitor the temperature to make sure it stays at 80-85°C.

5. While the water is heating, de-mold your samples prepared from last class. To help you keep track, record the colors of food dye used in part 1 below:

Control sample color: _____________

Stearic acid samples color: ___________

6. Now it's time to try to destroy your shape memory monsters. Bend each sample you prepared into a U-shape and clamp with the binder clip (see figure 2). Once the water has reached ~85°C, place the setup within the beaker, hanging the binder clips off of the metal spatula. Make sure all 3 samples are in the hot water all the way. (See Figure 3 for example setup.) Time for 1 minute.

7. Then using the spatula to help, remove the spatula from the hot water, and transfer the samples on the spatula directly into the room temperature water. Time for 1 minute.

8. Remove the samples from the room temperature water. Then remove the three binder clips.

9. **Did it work? Were your shape memory monsters deformed?** Write your observations below.

a) Control sample – containing no stearic acid:

b) Samples 2 and 3 – composites containing the stearic acid:

c) Do you notice any stearic acid on the surface of samples 2 and 3, or on your gloves? Is there any stearic acid in the water in the beakers? What does that suggest about the whether the stearic acid has chemically or physically combined with the silicone?

10. **If your composite materials have shape memory, then the monsters will come back to their original shape under the right conditions.** In order to test this, set 1 of your stearic acid samples aside, and place the **control sample and 1 of your stearic samples back in the hot water** (with no binder clips). Note: If the ends are stuck together, you may need to carefully tweeze them apart before placing in the hot water.

11. After 2 minutes, remove the samples from the hot water using the metal spatula to help.

a) What happened to the control sample?

Name: ______________________________________ Class period: ________

b) What happened to the stearic acid composite?

12. Repeat steps 6-11 for a second trial with your control sample and the stearic acid sample from step 11. (Keep your other stearic acid composite sample off to the side, and leave it there until the end of the lab.)

a) How did each of your samples perform in the second trial? Be specific about your observations at each point (hot water, cold water, then hot water again).

- Control Sample

- Stearic acid composite sample

13. Finally, take a look at the stearic acid sample that was **not** placed back in the hot water.

a) Did the shape memory monster recover on its own, without being put back into the hot water? ________

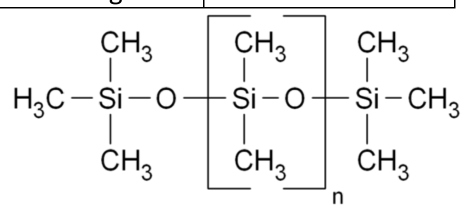
b) How was the *shape fixity* – in other words, did the sample start to flatten at all, or keep in the same c-shape?

14. Make sure the hot plate is turned off and unplugged. Clean up your lab area. Throw away the two stearic acid composite samples. You may take home the control sample, if you would like, or throw it away as well.

Post Lab Questions

Use the difference in electronegativity to calculate the type of bonds (ionic, polar-, or nonpolar covalent) contained within each compound.

Electronegativity Difference	Type of Bond
0.0 to ~0.4	Nonpolar Covalent
~0.4 to ~2.0	Polar Covalent
2.0 or higher	Ionic


[3]

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[3] Silicone structural image from https://chem.libretexts.org/Exemplars_and_Case_Studies/Case_Studies/Heat_and_Chemical_Resistant_Silicone_Rubber/Silicones_7%3A_Polymer_Chemistry%2C_Thermochemistry%2C_and_Kinetics

Name: ______________________________________ Class period: ________

1. Chemical composition of silicone: Note the central chain in the picture above would repeat **thousands** of times in a single silicone molecule.

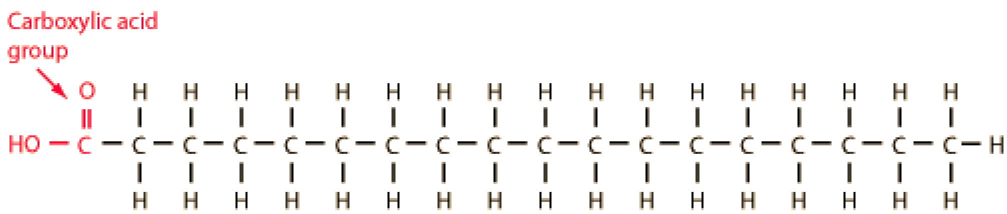
a) Bond type for Si - O

b) Bond type for Si – C

c) Bond type for C - H

[4]

2. Chemical composition of stearic acid:



Stearic acid, an example of a saturated fatty acid

a) Bond type for C – H

b) Bond type for C – O

c) Bond type for O - H

3. What intermolecular forces would apply to each compound?

a) Silicone

b) Stearic Acid

4. Based on the intermolecular forces, would you expect the stearic acid to dissolve in the silicone? Why or why not?

5. Draw a particulate diagram to illustrate what is happening at each point of the shape memory testing cycle.

a) Heating for 1 minute w/binder clip b) Cooling down for 1 minute w/binder clip c) Re-heating for 1 minute

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[4] Stearic acid structural formula from http://hyperphysics.phy-astr.gsu.edu/hbase/Organic/fataci.html