

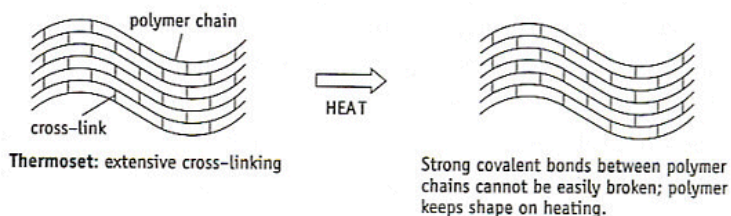
Shape Memory Composite Lab – Teacher Notes

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 metal tire that will repair itself at the right temperature – that’s something NASA scientists and engineers are working on right now for the Mars Rover¹. Other current scientific research is being done on **shape memory materials** made from polymers.

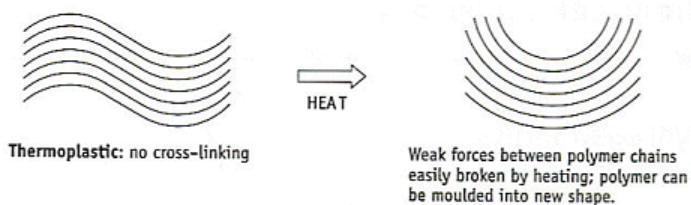


Recall that **polymers** are long chain-like molecules. These polymer chains can become **cross-linked**, a bit like a tangle of necklaces that have formed several knots. Each crosslink connects the strand to other strands, causing the tangled polymer chains to move together when a force is applied. 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 silicone rubber. The thermoset material provides the permanent shape, 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.

Pre-Lab Questions:

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: _____

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

- *Shape memory materials can be programmed to a specific shape, de-formed, and then return to their original shape under the correct conditions.*

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

- *More crosslinking makes the material more durable and extremely heat resistant.*

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

- *Thermoplastic materials have little to no crosslinking. They can be easily melted and re-formed into new shapes. If performing the demo, poly doh is an example of a thermoplastic material.*
- *Thermoset materials have strong crosslinking. This makes them good for high temperature applications; they are also stronger and more durable.*

4. What is a composite material?

- *Composite materials are mixtures that combine the physical and chemical properties of different materials to create unique properties. Concrete is an example of a composite material. Wood and bones are natural composites.*

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

- *Composite materials are developed to take advantage of varying properties of their components. Concrete is much stronger than any of its components (paste, sand, gravel, or water) by itself. Silicone does not have shape memory by itself. Neither does stearic acid.*

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 – can be purchased on Amazon.com and from Smooth On directly – 1 trial kit contains enough for roughly 85 to 90 lab groups
- 4 small cups (2 or 3 oz. size works best)
- Metal spatula
- Paper towels
- 3 small plastic molds for candy-making

TEACHER NOTE: *The quantities are for [these Halloween molds](#) from Amazon.com, but any plastic molds will work. Just make sure **not to use silicone molds**, or the samples will bond to the molds. The quantities needed in this lab depend on your plastic molds. If using a slightly larger mold (such as these 1/8" depth [snowflake molds](#)), then use 5-6 grams each of parts A and B and around 1.0 to 1.4 grams of stearic acid. Note: too much stearic acid can make the samples difficult to de-mold.*

Making Shape Memory Monsters - Procedure

See student version for detailed procedure for part 1.

Name: _____

Class period: _____

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?

- *Heterogeneous – the stearic acid is clearly visible in the mixture*

2. What kind of mixture (homogeneous or heterogeneous) was the combination of A and B initially (no stearic acid)?

- *Homogeneous – parts A and B can no longer be distinguished once they have mixed.*

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”?

- *A chemical reaction is occurring. After 4 minutes, the chemical reaction between parts A and B has reached the point where enough bonds are formed that re-molding is difficult.*

Lab – Part 2 – Lab – Part 2 – Fighting the Shape Memory Monsters

Testing the Shape Memory Properties of your Material

Lab Part 2 Materials: (for each lab group)

- Safety glasses or goggles
- Safety gloves
- 2 large beakers (400 mL or larger)
- Hot plate
- Thermometer

- Metal spatula
- 2 large 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.

3. Place one of the beakers on the hot plate, and turn on high. Put in a thermometer to observe the temperature. Heat until $\sim 85^{\circ}\text{C}$. You don't want the water to boil, but it needs to stay above the melting point of stearic acid, which is $\sim 70^{\circ}\text{C}$. 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 $80\text{-}85^{\circ}\text{C}$.



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



help

Name: _____

Class period: _____

Control sample color: _____

Stearic acid samples color: _____

6. Bend each sample you prepared into a U-shape and clamp with the binder clip (see figure 2). Once the water has reached $\sim 85^{\circ}\text{C}$, place the setup within the beaker, hanging the binder clips off of the metal spatula. Make sure both 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 two binder clips.

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

a) Control sample – containing no stearic acid:

- *The control sample should not change shape when heated, when cooled, or heated again. Silicone is a thermoset material by itself, and will not display any shape memory.*

b) Sample 2 – composite containing the stearic acid:

- *The sample containing shape memory should display shape memory. When it is heated past the melting point of the stearic acid, the crystals melt allowing the sample to be bent by the binder clip. The cooling allows the crystals to re-form, fixing the sample in the C-shape.*

c) Do you notice any stearic acid on the surface of sample 2, 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?

- *Typically, some of the stearic acid escapes from the silicone thermoset network during the testing procedure. Stearic acid is not soluble in water, so it will be visible on the surface of the water after the test is complete. The stearic acid has only physically combined with the silicone, so the testing often causes some to be removed from the surface of the sample.*

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.

Teacher Note: In order for a material to have shape memory properties, it must be able to hold a new shape after being heated & cooled to recrystallize the crosslinks. Then, the material must be able to reset to its original shape configuration when re-heated. This step tests whether the material recovers to the original shape.

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

a) What happened to the control sample? *Nothing happens to the control sample. It stays exactly the same.*

Name: _____

Class period: _____

b) What happened to the stearic acid composite? *The composite material should recover back to its original shape, or very close to it.*

12. Repeat steps 6-11 for a second trial.

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

Results are usually the same.

- Stearic acid composite sample

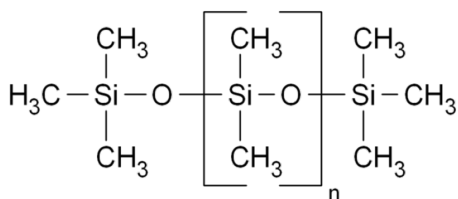
Results are usually the same.

13. Clean up your lab area. Make sure the hot plate is turned off and unplugged.

- *Helpful Hints: For clean-up, stearic acid is soluble in alcohols. A wash bottle with isopropyl alcohol and a paper towel works best on the beakers; heating slightly on the hot plate will also increase the solubility. You may want to have all of the classes complete the testing using the same beakers, without worrying overly about cleaning up the stearic acid until the end. The stearic acid remaining in the beaker will not affect the results of the lab.*

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
~0.41 to ~2.0	Polar Covalent
2.0 or higher	Ionic

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

- a) Bond type for Si - O ~1.7 = *Polar covalent*

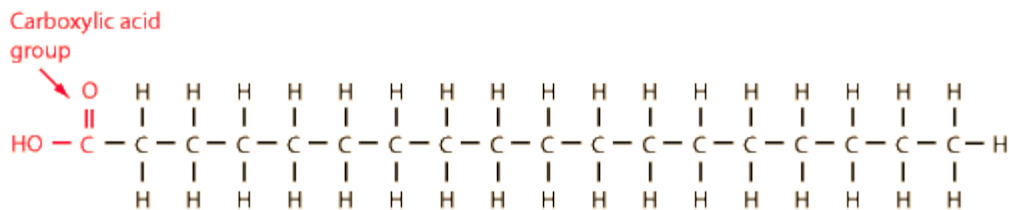
Name: _____

Class period: _____

b) Bond type for Si – C $\sim 0.7 = \text{Polar Covalent, but less so than Si-O}$

c) Bond type for C – H $\sim 0.4 = \text{nonpolar covalent}$

2. Chemical composition of stearic acid:



Stearic acid, an example of a saturated fatty acid

a) Bond type for C – H

$\sim 0.4 = \text{nonpolar covalent}$

b) Bond type for C – O

$\sim 1.0 = \text{polar covalent}$

c) Bond type for O – H

$\sim 1.4 = \text{polar covalent}$

3. What intermolecular forces would apply to each compound?

a) Silicone *Dipole-dipole interactions along the inner chain, but mainly London Dispersion (van der Waals) interactions due to the molecule length and the methyl groups*

b) Stearic Acid *Almost entirely London Dispersion (van der Waals) interactions, but one polar group on the end with Hydrogen bonding and dipole-dipole*

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

- *Students will most likely argue yes, because of the significance of the London Dispersion interactions in both molecules.*
- *When discussing as a class, remind students that the stearic acid does not dissolve in the silicone at room temperature. This is a great introduction to discussing the complexity of other factors that can affect solubility such as the phase of each material, the temperature, and the shape of the molecules involved.*

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

▶ Testing the shape memory cycle :

1-2. Heating the material under the stress of the binder clip, until it's above the melting point of the stearic acid (~70°C). This will allow the crystals in the composite network to become liquid and help the polymer chains to move and bend into the new shape

3. Cooling the material, so that the stearic acid recrystallizes -- locking the material into the new shape

4. Removing the stress of the binder clip to see if the material has shape fixity (does it stay in the new shape created from the binder clip?)

5. Finally, re-heating it above the melting point of stearic acid without any stress, to see if it can recover to the original shape held by the silicone thermoset network (shape recovery)

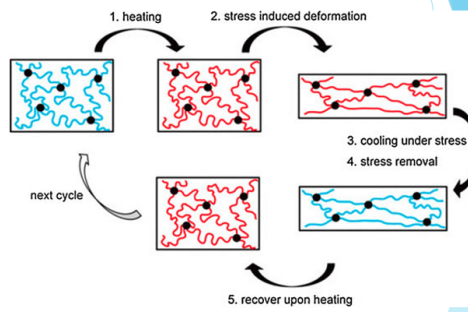


Image from https://www.researchgate.net/figure/The-molecular-mechanism-of-a-dual-shape-memory-polymer-throughout-a-thermal-cycle-Black_fig4_306334643