Growing Crystals: Hot Ice Activity
Participant Guide

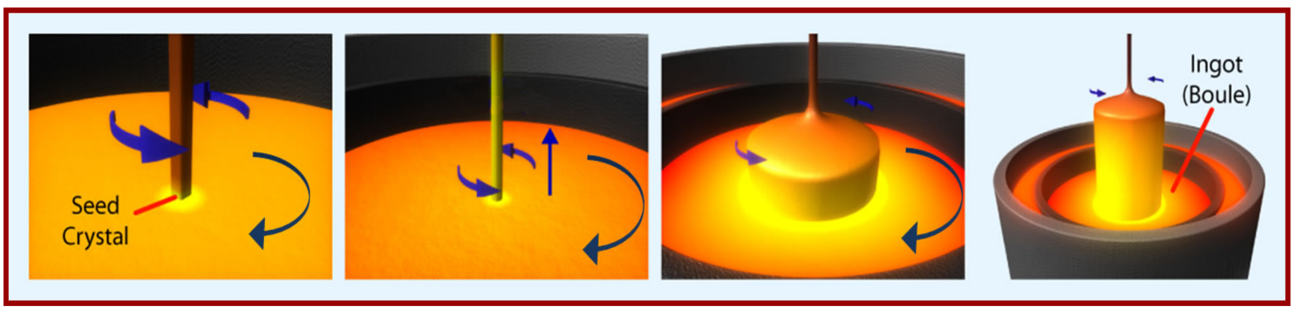
Description and Estimated Time to Complete

In this activity sodium acetate trihydrate is used to demonstrate how crystals grow from a seed crystal. Using a hot plate, you will dissolve polycrystalline sodium acetate trihydrate in a beaker, creating a supersaturated liquid solution. Once the solution cools, you will trigger crystal growth by placing a seed crystal in the supersaturated sodium acetate trihydrate solution.

Estimated Time to Complete
Allow at least one hour to complete this activity.

Introduction

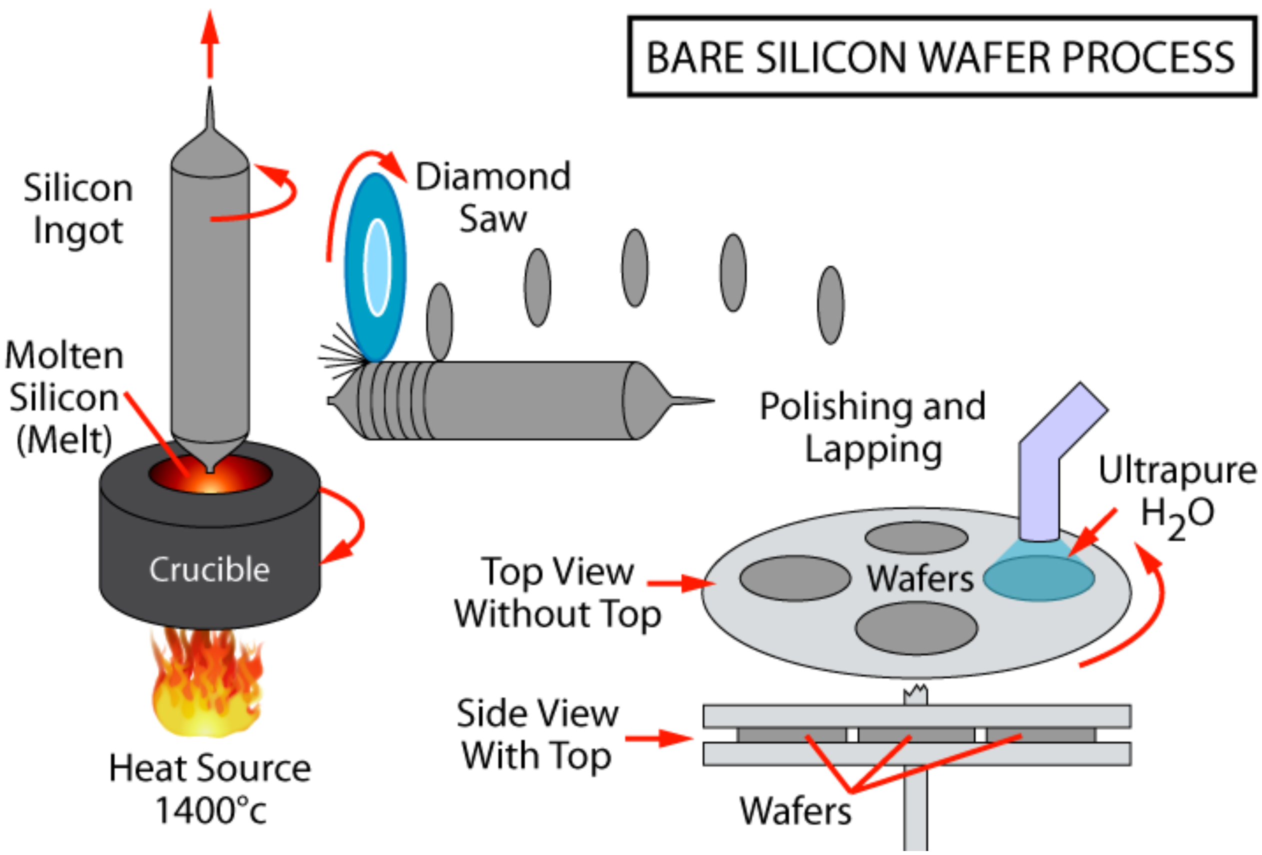
MEMS (microelectromechanical systems) are fabricated using monocrystalline silicon wafers. The wafers are cut from a silicon ingot that is formed by melting chunks of polycrystalline solids in a large crucible. Once melted a "seed crystal" is placed in the liquid silicon to stimulate crystal growth for a specific crystal orientation. Over several hours a long ingot of pure monocrystalline silicon is slowly pulled from the melt. Below are the steps for "growing" this monocrystalline ingot.



1. First we start with very pure polycrystalline silicon material (99.999999999% pure!)
2. The pure silicon is melted in a crucible at 1425°C. (This molten silicon is called "the melt".)
3. A seed crystal is precisely oriented and mounted on a rod then lowered into the melt *(left image)*. Silicon atoms in the melt align to the same crystal orientation of the seed.
4. As the seed crystal is slowly pulled out of the melt, the seed and the crucible are rotated in opposite directions A large crystal ingot or boule is formed by controlling the temperature gradient of the melt, the speed of rotation, and the rate of the pull of the rod. The slower the "pull", the larger the diameter of the crystal ingot that forms. (This process can take several days to complete and is called the Czochralski (CZ) Method of growing silicon.)

The seed crystal acts as a nucleation site for the alignment of the atoms in the molten silicon. The alignment of the seed crystal relative to the melt determines the orientation of the subsequently grown silicon crystal ingot. The wafers cut from this crystal maintain this orientation.

Once the ingot is formed, it is removed from the equipment, then ground down to form a smooth cylinder with a consistent diameter from end to end. The ingot is now cut into silicon wafers using a diamond saw or diamond coated wires. The individual wafers are polished for a smooth, uniform surface. This process is called "polishing and lapping".



In this activity you will simulate the crystal growing process by melting sodium acetate trihydrate then stimulate the growth of a crystal by exposing "the melt" to a seed. Two types of reactions occur during this activity – endothermic and exothermic.

Endothermic and Exothermic Reactions

The melting of the sodium acetate trihydrate to form the supersaturated solution and subsequent re-crystallization demonstrates both endothermic and exothermic reactions.

Endothermic - As the sodium acetate trihydrate solid is heated above its melting point of 58°C it absorbs heat energy (an endothermic reaction). Continued heating allows for *all* of the sodium acetate trihydrate to dissolve. This process results in an unstable supersaturated solution.

Exothermic - When one initiates recrystallization of the supersaturated solution, the system crystallizes or freezes (forms a solid) between 54-58° C, giving off heat energy (an exothermic reaction).

Below is how this reversible reaction is notated. The left side of the equation is the liquid state (*l*) and the right side is the solid state (*s*). When reading right to left, heat is added to the solid state resulting in a liquid. Reading left to right, when the supersaturated liquid solution crystalizes, a solid forms giving off heat.

$$NaC2H3O2{\cdot}3H2Ol \leftrightarrows NaC2H3O2{\cdot}3H2Os + 19.7kJ/mole \text{ (heat)}$$

In this experiment the dissolved supersaturated solution of sodium acetate trihydrate (the liquid) cools below room temperature to about 15°C (59°F). When this temperature is reached a seed crystal is added causing the liquid solution to rapidly crystallize or freeze, releasing its heat (*left to right in the preceding equation*). Once crystallized, the heat can be felt through the beaker by holding the beaker in the palm of the hand. This exothermic reaction of the sodium acetate trihydrate gives off enough heat to reach its freezing point of 58°C (136°F) which is hot enough to cause burns; therefore caution is recommended.

Activity Objective and Outcomes

Activity Objective

Demonstrate the growth of a crystal by heating sodium acetate trihydrate below its melting point to form a supersaturated liquid solution, and then initiating crystal growth by placing a "seed crystal" into the liquid.

Activity Outcomes

This activity has two possible outcomes. One is the formation of a solid crystal by placing a "seed" into the dissolved sodium acetate trihydrate. The other outcome is to form a crystal structure by pouring the liquid sodium acetate trihydrate into a flat container or watch glass containing a seed crystal.

Resources

NurdRage "How To Make Hot Ice – The Complete Guide" Uploaded Oct. 17, 2009
https://youtu.be/Yxk3OZWq9Ls?list=PLFZzcUkE9mZVGxkdSq0xlY9kleXDx6wyT

https://www.flinnsci.com/media/621073/91433.pdf

Fisher Scientific Material Safety Data Sheet (MSDS) for Sodium Acetate Trihydrate

Supplies (*Needed for each team. The other items can be shared)

50 grams of 100% Sodium Acetate Trihydrate*
200ml beaker*
Watch glass*
3 ml plastic pipette*
Beaker tongs
Hot plate
Weigh boat
Small pitcher (for distilled water)
Weighing scale (grams)
Stainless steel chemistry scoop*
Glass Casserole dish used for the Cooling Bath
Distilled water for adding to the sodium acetate trihydrate
Tap water for the cooling bath
Thermometer

Personal Protective Equipment (PPE)

- Gloves
- Safety Glasses/Goggles

Activity: Growing Crystals – Hot Ice

<u>Procedure:</u>

Procedural Notes:

Read these procedural notes and the entire procedure before starting this experiment.

a. The more sodium acetate trihydrate crystals you use the longer it takes to dissolve and cool. By using 50 grams, you can keep the time of this experiment to less than an hour.
b. The addition of water is to help speed up the dissolution of the sodium acetate trihydrate crystals. The sodium acetate trihydrate crystals can dissolve without adding water because they have three water molecules attached; however, NOT adding water would slow down the dissolution, adding to the overall time to do this experiment.
c. Once the sodium acetate trihydrate is in liquid form you may notice crystals forming on the stainless steel (SS) scoop that you use to stir the solution. This crystal formation indicates that the sodium acetate trihydrate is NOT completely dissolved. Once you see no crystal formation on the SS scoop when stirring, the sodium acetate trihydrate is completely dissolved.

Procedure Steps

1. Locate the Fisher Scientific Material Safety Data Sheet (MSDS) for sodium acetate trihydrate. (Do an online search) Review the MSDS and answer the following questions before starting this experiment.
   a. What is the chemical formula (molecular formula) for sodium acetate trihydrate?
   b. What are the potential acute health hazards of working with sodium acetate trihydrate?
   c. At what temperature (°C and °F) will sodium acetate trihydrate auto-ignite?
   d. What is the melting temperature for sodium acetate trihydrate?
   e. What are the handling requirements for sodium acetate trihydrate?
   f. What PPE (personal protective equipment) should one wear while doing this activity?
   g. What personal precaution should one take while doing this activity?
2. Turn on the hot plate to high.
3. Using the scoop and the weigh boat, weigh out 50 grams of sodium acetate trihydrate. Transfer to the 200 ml beaker.
4. Place the beaker with sodium acetate trihydrate on the hot plate.
5. Using the stainless steel chemistry scoop, stir the crystals frequently to distribute the heat evenly. The sodium acetate should start to dissolve. Sodium acetate trihydrate dissolves at 58°C.
6. As the sodium acetate trihydrate dissolves, crystals start to form on the sides of the beaker. Fill the pipette with 2 ml of distilled water and rinse off the crystals as they appear. At this point you can reduce the temperature of the hot plate to a medium high.
7. If the liquid starts to cloud or crystallize, add more liquid, 1 ml at a time, until the liquid clears.
8. While the sodium acetate trihydrate is dissolving, prepare the cooling bath by adding cold tap water to the glass casserole dish, about 2 inches deep in the dish.
9. Once the crystals have dissolved into a clear liquid, check the solution to ensure that it is "completely" dissolved by stirring the liquid with the SS scoop. If any crystals form on the scoop, then the sodium acetate trihydrate is NOT completely dissolved. Continue to heat. The sodium acetate trihydrate is completely dissolved when no crystals form on the SS scoop.
10. Once completely dissolved (no clouding or crystal formations), use the beaker tongs to remove the beaker from the hot plate and place in the cooling bath. Cover the beaker with a watch glass to prevent premature crystallization of the sodium acetate trihydrate.
11. Feel the sides of the beaker. When it feels cool to the touch and below room temperature, check the temperature with the thermometer. ***WARNING**: Before checking the temperature, thoroughly clean the tip and surfaces of the thermometer to prevent premature crystallization. Any dirt or debris will become a nucleation site for crystal formation.*
12. **Once the solution reaches 15°C**, extract a seed crystal (the size of a dust particle if possible) from the bag of sodium acetate trihydrate. Perform one of the following:
    a. Drop the dust size crystal of sodium acetate trihydrate into the beaker. This seed crystal initiates crystallization. You should see a crystal forming in all directions starting at the seed. *(Suggestion: Take a video or pictures of the crystal formation.)*

b. Place the dust size seed crystal on the watch plate. Slowly pour out the dissolved sodium acetate trihydrate into the watch glass on top of the seed. As you pour, the sodium acetate trihydrate should crystallize into a stalagmite formation. *(Suggestion: Take a video or pictures of the crystal formation.)*

Note: You can reuse crystallized sodium acetate trihydrate. Return the beaker to the hot plate and slowing reheat, adding distilled water if needed. You can continue to reuse the solution until it becomes contaminated.

Post-Activity Questions

1. Which procedure did you perform (12a or 12b)?
    a. Explain exactly what you saw.
    b. In your own words, describe the crystallization process that you observed.
2. Why does sodium acetate trihydrate work for this experiment?
3. What are the melting point and the freezing point of sodium acetate trihydrate?
4. What is supercooling?
5. What is the purpose of the seed crystal?
6. What are two applications for sodium acetate?

Support for this work was provided by the National Science Foundation's Advanced Technological Education (ATE) Program through Grants. For more learning modules related to microtechnology, visit the SCME website (http://scme-nm.org).