

## **Crystal Study**

### **Instructor Notes**

#### ***Reliability***

This experiment consistently works. The only drawback is the possibility of breaking the glass squares by dropping them or when dismantling the glass-stuff-glass “sandwich” while the stuff is still molten (easily repaired though).

#### ***Estimated Time for Activity***

One class period.

#### ***Teacher Tips***

1. The purpose of this lab is to provide students an opportunity to 1) observe macro crystalline growth, 2) observe how crystals grow together and form grain boundaries, and 3) compare various kinds of crystals. This knowledge can then be transferred to metals and ceramics, which form crystalline structure in the same manner as observed except on a micro scale.
2. It is recommended that the teacher prepare the glass slides for this activity. The instructions for doing this are given below. The student activity includes only the procedures for warming and cooling the plates once they are made. Once these glass sandwiches are made, they can be re-used many times and stored from year to year.
3. Suggested materials that may be used to crystallize include the following:
  - phenylsalicylate, also called salol
  - thymol, strong “listerine” odor
  - benzoic acid
  - urea, crystal growth is very small, but large grains appear, (teaches grain boundaries and grains)
  - naphthalene
  - naphthol (also may want to use magnification to study crystals and boundaries)
  - p-dichlorobenzene (fumes).

4. Suggested materials which will make an amorphous structure as a comparison to the crystalline materials:

- paraffin
- stearic acid

**Note:** No crystals form with these chemicals.

5. To make glass plate sandwiches

- Put plates on cold hot plates and warm them slowly.
- Sprinkle a few grains of chemical on the plate, and allow them to melt. Form a small puddle of molten chemical in the center of the plate.
- Take a second warm plate, and carefully cover the puddle. To reduce air bubbles, place the top plate on edge at one end of the bottom plate and slowly lower it to cover the molten chemical evenly and force the air out the sides.

**Caution:** If the chemical is forced from between the two glass plates, it can drip onto the hot plate and cause smelly or harmful fumes to volatilize.

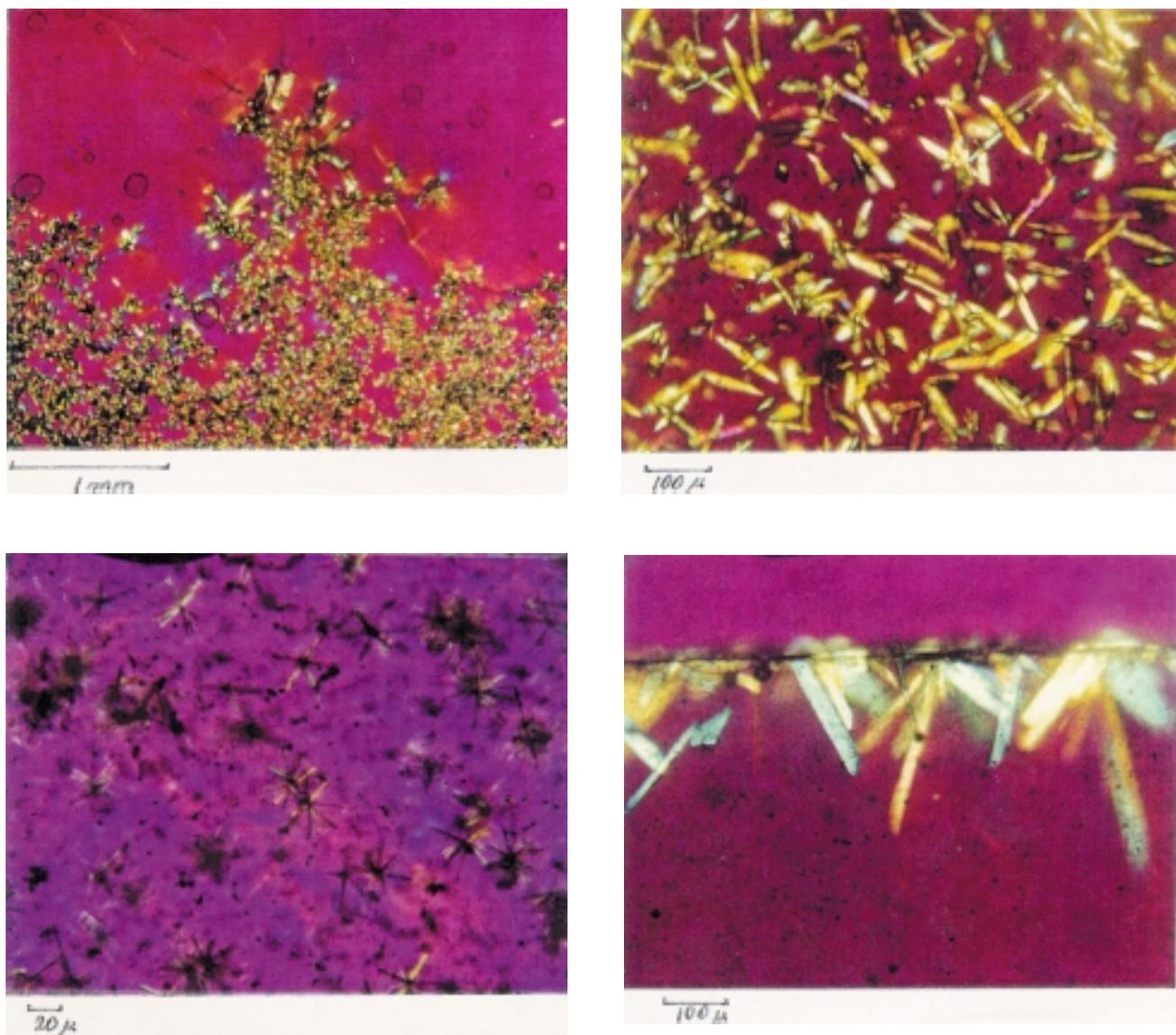
- Allow the sandwich to cool slowly.
- Make a third plate, which is a mixture of two chemicals (make sure they are compatible i.e., naphthol and naphthalene are okay), and observe how the mixture behaves under slow and rapid cooling. A series of three plates can be made with different concentrations of the two chemicals.
- Cool the plates under a gradient. Insulate one-half of the plate so it cools more slowly than the other half. Compare the slow and fast cool regions and the transition zone. Note the shape and size of the crystals relative to how the plate was cooled. Repeat this with the plates that have two chemicals mixed on them.

6. Why does crystal growth matter? Many people and industries rely on controlled crystal growth. The manufacturers of sugar and salt, for example, must produce uniformly sized grains tons at a time. They need to know about how quickly the grains grow and how to make them all the same size. You may want to compare your crystals grown from molten solutions to those made from aqueous solutions.

Drug companies also care about crystallization. Crystallization can be used to purify chemicals. Think about the plates that had a mixture of chemicals on them. Can you think of a way to "unmix" them first by heating and cooling?

The structure of a single crystal can be used to help identify the compound.

How would you make large single crystals for further analysis? (see Figure 4.3 attached.)



**Figure 4.3.** Varied Crystals Growing in Chemical Solutions

## Notes on Polarized Light

1. Works very well when observing transparent materials that are crystalline or have areas of stress.
2. Polarized light is used a number of ways in MST. This activity is an introduction to the concept. It provides background information that can be used in the Crystal Study Lab and for making glass (Ceramics section).
3. Light travels as a wave, much like sound. If you were to look at a light wave as it travels along, you would see a regular rise and fall in intensity and a constant distance between peaks and troughs, commonly known as a sine wave. (See Figure 4.4).

If you were to look at a bunch of light waves end-on, they would look pretty messy. This is because the waves are not all in the same plane. The general effect would be something like Figure 4.5.

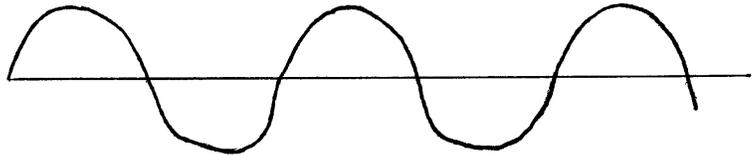


Figure 4.4.

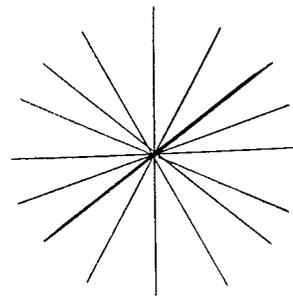


Figure 4.5.

4. If we could get some light waves all lined up in the same direction, some neat things could be done with them. This effect can be accomplished by using a polarizer, which is a material that looks like a picket fence to a light wave. Only the light waves oriented parallel to the picket fence are able to pass through it, and if a second polarizing filter is held with its axis perpendicular to the first, no light can pass through the pair.
5. Polarizing films can be made by heating a sandwich made from needle-like crystals of iodoquinine sulfate between two sheets of plastic. As it cools, the plastic sheets are pulled in the opposite direction to line up the crystals. This alignment produces the picket-fence effect as described above.

6. Light that is reflected from shiny surfaces is polarized to some extent. That is why polarized sunglasses work to cut glare, because the lenses in these glasses are oriented to filter out much reflected light. These lenses can be taken apart and used as a source of polarized film for the experiments involving stress visualization or crystallization.
7. In this handbook, polarized light is used to observe two phenomena, crystal orientation and stress. In some crystalline materials, polarized light is transmitted in some directions better than in other directions. This difference in crystal orientation can be observed when viewing these crystals between cross-polarized film. This process is explained in this experiment.  

Polarized light also interacts with certain transparent materials under stress. As the polarized light passes through the area of stress, it is slightly rotated causing color bands to be generated, which can be observed through another polarized film. This process is used to observe stress in transparent glass and plastics as discussed in the Ceramics section of this handbook.
8. Reference: Wood, E.A. 1964. *Crystals and Light, An Introduction to Optical Crystallography*, Van Nostrand, New York.
9. Devise a way to measure the crystals, and have students put a scale in their sketches.
10. How does polarized light work?
11. What other materials can be analyzed with polarized light?

### ***Extension Activity***

1. This would be an opportune time to invite a "rock hound," jeweler, geologist, mineralogist, or earth science teacher to visit your classroom to discuss and show various crystalline materials.
2. Other variations to this lab follow.
  - a) As the glass slides are cooling, and crystals have not yet formed, place an aluminum rod on the top glass plate. The aluminum acts as a heat sink. Observe how this changes previously run samples.
  - b) Have a sample in a glass sandwich where one surface of the glass is scratched. Observe how this scratch affects results of crystal growth.
  - c) On a single plate of glass, melt a small pool of suggested material. As this molten pool cools, drop a small "seed crystal" of the same material into it. Observe how the seed crystal affects crystal growth.

3. Examine the plates using transmitted light and a microscope or hand lens.
4. Have students practice sketching what they see in their journals. Have them pay special attention to how “regular” the crystals are. They are easy to draw because there are so many straight lines. Why is this? Before the structure of the atom was known, early mineralogists thought crystals were made of building blocks. What shape blocks would you need to make your crystals? How thick are they?

Compare your flat plates to salt, sugar, or even rock candy. See if you can get the students thinking about crystals as three-dimensional objects.

If you live in an area with cold winters, discuss or examine frost, snowflakes, ice, and the variety of shapes crystalline water can take. It’s all water! Guess the shape of the building block.

### **Safety**

1. Use Pyrex or other thermal shock-resistant lab glass for glass plates. Thin plates (1/8 in. or less) are better than thicker plates. Microscope slides work well.
2. *Prepare these plates in advance.* This is most easily done by placing the glass plate on a hot plate set on low temperature in the hood, melting the chemical on the warm glass plate, and placing the second plate on top. To reduce breakage of the glass plates, warm all the plates slowly on the hot plate, and handle them with tweezers.
3. You need to consider ventilation. Some substances produce irritating fumes; others produce slightly toxic fumes or fumes that will make many light-headed. Note: thymol, naphthalene, and naphthol produce fumes and strong odors. Benzoic acid has very irritating fumes, phenylsalicylate has toxic fumes. **Use these chemicals cautiously!** Don’t spill them on the hot plate’s surface. Handle only small quantities around the hot plate.
4. Hot plates can cause burns.

## **Activity: Crystal Study**

### ***Student Learning Objectives***

At the end of the activity students will be able to:

- follow a procedure that allows the study of crystal growth, size, and shapes
- demonstrate their observation and recording skills through journal writing and discussion.

### ***Materials***

See Instructor Notes

### ***Equipment***

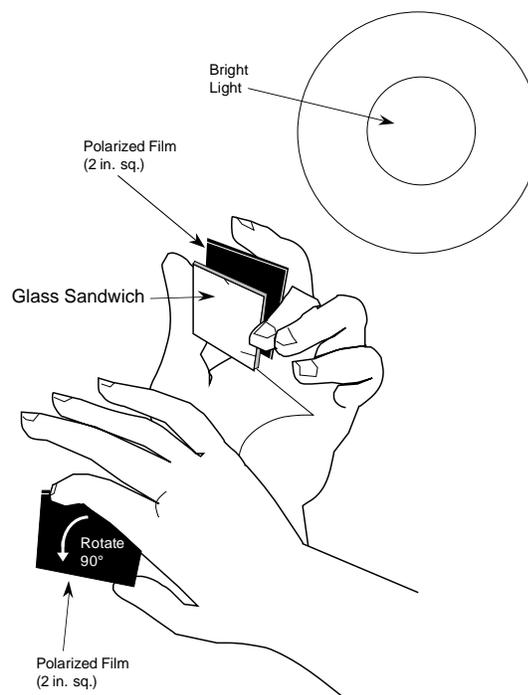
- Prepared glass plate sandwiches, 5 cm x 5 cm, 2 ea.
- Colored pencils
- Polarized film, 5 cm x 5 cm
- Magnifying lens
- Hot plate
- Tweezers/tongs

**Caution:** Do not overheat glass plates. Do not put your head directly over the hot plate when heating chemicals. Avoid breathing fumes.

### ***Procedure***

1. Obtain two different glass plate sandwiches, one marked "A," the other marked "B."
2. Obtain two pieces of polarized film.
3. Place glass "A" on a cold hot plate and turn on hot plate to a low setting ("2" is typical). (DO NOT TURN TO A HIGHER SETTING.)
4. Carefully observe the crystal formation between the two pieces of laminated glass. When the crystals START to melt, immediately remove the glass from the hot plate using tweezers. Turn off hot plate. Continue observations.
5. Record observations in your journal. Include sketches of crystals.

- Place glass between polarized film with a light source behind it. Turn or rotate pieces of the film 90° and observe (see Figure 4.6). The two pieces of film can be arranged to either transmit or block light. View the glass plate with the film in both positions and without the film. Illustrate what you see in your journal using, if needed, colored pencils.



**Figure 4.6.** Use of Polarized Film for Analyzing Crystal Structures

- Repeat steps 3 - 6 using glass "B."
- If time allows, repeat steps 3 - 7, using samples with different chemicals.

### ***Additional Activities***

- Cool the plates slowly, and sketch what you see.
- Cool the plates rapidly, and compare to the slow-cool method.
- Observe other materials and light sources using the polarized film to better understand polarized light.