

Crystallization in Sugar Glass and Its Melts - Low Cost Experiments in Glass

Sarah Horst* and William Heffner, IMI-NFG, Lehigh University, Bethlehem, PA

Objective:

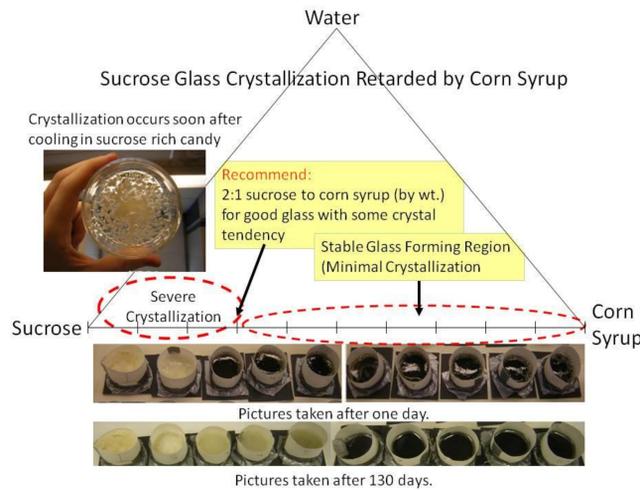
As part of the IMI's series of low cost experiments with glass & material science, we have also developed several hands-on experiments associated with crystallization. Sugar glass provides a convenient system to study crystallization. We present here experimental methods to investigate several aspects of crystallization in this accessible system, including low-cost, student-built apparatus. These methods provide quantitative results illustrating on important principles of crystallization and growth.

The primary focus here includes experiments on:

Surface crystallization at room temperature

Bulk crystallization at elevated temperatures

Sucrose & water provides a simple, convenient glass forming system for student experiments. However the simple binary is very prone to crystallization. The addition of corn syrup (primarily glucose with some higher length dextrans) reduces the tendency to crystallization. See the figure to right.



Excellent for Crystal Growth Studies

Two Distinct Mechanisms

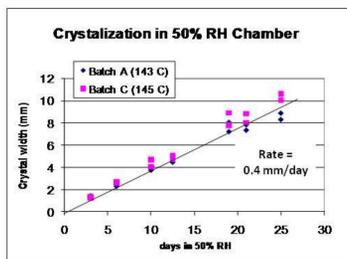


Have quantitative experiments for both

Surface Crystallization

Crystal Growth at Candy Surface

Moisture mediated surface crystallization at Room Temp



Surface Crystallization is Very Humidity Dependent:

- Dry: No crystal growth observed after several months
- 50% RH: Good, uniform crystal growth (= 0.4mm/day)
- 100% RH: Outer edge becomes fluid from absorbed water

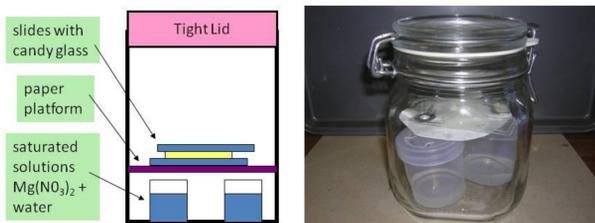
Procedure:

- Samples prepared by placing a few drops of sugar glass between two glass slides, held apart by a space (washer)
- Samples placed in constant humidity chambers (dry, 50% RH, 100% RH)
- Every few days remove and photograph samples
- Measure the width of the crystal ring on the photo and record



Applicable for high school science project

Student Humidity Chamber



Humidity Chamber:

- For 0% (dry): use CaSO₄ as a desiccant
- For 50% RH: use saturated solutions of Mg(NO₃)₂ + water
- For 100% RH: Use water or wet cotton

Conclusion:

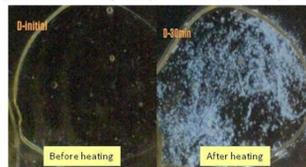
We have developed low-cost experimental methods for investigating both surface and bulk crystallization. These methods provide quantitative results illustrating several important aspects of crystallization and growth, such as the linear growth rate of surface crystals at moderate humidity and a maximum crystal growth rate at 120 C. More importantly, these results and procedures are open-ended and enable the student to further investigate this interesting system. We are enthusiastic about further explorations of crystal morphology vs. temperature. For additional details and future updates please see our education page at: www.lehigh.edu/GlassEducation.htm

Bulk Crystallization

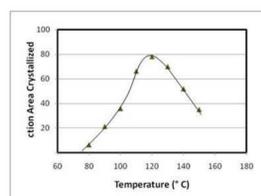
Crystallization from Bulk in Molten Solution

Finding the maximum crystal growth temperature

Crystals form slowly in liquid at elevated temperature



Photomicrographs taken from QX5 microscope through crossed polarizers



Procedure:

- Heat samples to temperature from 80°C - 150°C in intervals of 10°C and leave at temperature for 20 minutes.
- Remove sample and photograph crystallization through polarizers.
- Analyze the images to determine the area fraction of crystals using Image J freeware.

Repeatability

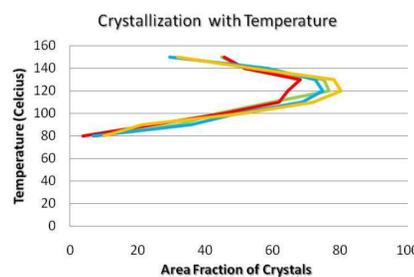
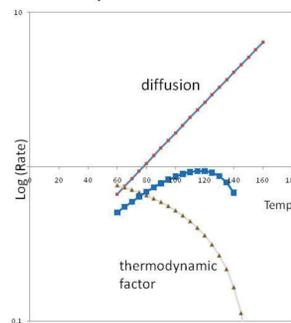


Figure above shows results for four different sets of samples.

Simple Model for Crystallization Rates



Rates depend on two activation steps:

- Change in free energy between phases (ΔG energy barrier)
- Diffusion of atoms to rearrange into a crystal
- Both are very temperature dependent
- Actual curve for rate is the product with maximum below T_m .

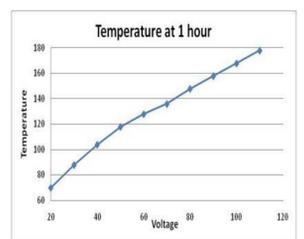
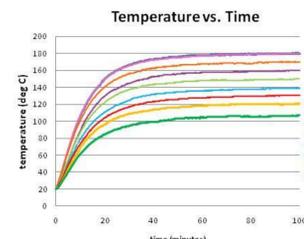
Student can use simple Excel model their fit the observed data!

Equipment



Oven + dimmer cost = \$20

Temperature uniformity $\pm 1^\circ\text{C}$ typical



QX5 Computer Microscope: \$75



Samples: \$5



Desiccator: \$5

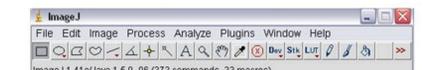
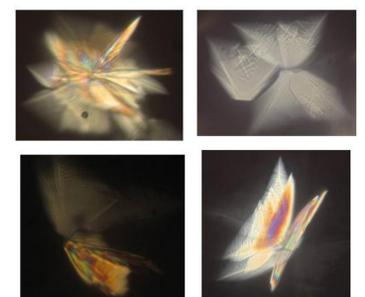


Image J Analysis Software: \$ Free
From NIH at: <http://rsbweb.nih.gov/ij/>

Crystal Morphology



An interesting range of crystal morphologies were observed under higher magnification through our home-built hot stage. Levenson and Hartel reported some of the same morphologies in their 2004 paper. Ref: D.A. Levenson, R.W. Hartel, *Journal of Food Engineering*, "Nucleation of amorphous sucrose-corn syrup mixtures"